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FINAL REPORT
THERMIONIC CATHODE EVALUATION STUDY

RAYTHEON COMPANY
Microwave and Power Tube Division
Waltham, Massachusetts

FINAL REPORT
THERMIONIC CATHODE EVALUATION STUDY

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ABSTRACT

Raytheon's Materials and Techniques Group has conducted a study of three different cathode types for the Jet Propulsion Laboratory with the objective of attaining thermionic emission currents of 1.0 A/cm^2 under dc voltage conditions at the lowest possible cathode temperature.

The pore type dispenser cathodes have been operating for at least 16,502 hours at current densities varying from 0.2 to 1.6 A/cm^2 and the cathode temperature varying from 950°C_{BR} to 1100°C_{BR} with no failures.

The standard oxide cathodes have operated from 13,925 to 15,678 hours on life burning at current densities of 0.075 to 0.6 A/cm^2 with the cathode temperatures varying from 800°C_{BR} to 850°C_{BR}. Slumping emission is noted during life at conditions above 0.15 A/cm^2 and 800°C. The emission slump increases with temperature up to 850°C and current loading up to 0.6 A/cm^2. Two emission failures are noted at 850°C (0.3 and 0.6 A/cm^2) at 10,000 hours of life. The coated particle cathodes showed the capability of being operated up to 0.55 A/cm^2 at 900°C. A comparison of the standard oxide and coated particle cathodes showed the pulsed emission peak current equal at 1.0 A/cm^2 and 850°C_{BR} cathode temperature.

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1.0 INTRODUCTION

The Materials and Techniques Group of Raytheon's Microwave and Power Tube Operation has conducted a study of the life burning capabilities of three different types of thermionic emitters for the Jet Propulsion Laboratory, California Institute of Technology.

This final report is a presentation of the work completed during the period from 1 January 1967 to 30 September 1969.

Three different electron tube cathodes were selected for evaluation of their thermionic properties and life burning capabilities for this program:

a. Pore-type dispenser cathode
b. Standard barium oxide cathode
c. Coated particle cathode

A life test vehicle was designed, fabricated, exhausted and tested electrically for conformance with the required current densities as specified for this study.

The diode test vehicle is a glass-enclosed test structure using plane parallel geometry with a viewing port in the anode for determination of cathode temperature.

A total of 166 diodes containing the three cathode types were constructed, exhausted and tested for this program.

A total of 50 diodes, using pore-type dispenser, standard barium-strontium oxide and coated particle cathodes, was selected for life burning.

This report will describe the objectives of this study and the selection of test diodes for life test burning, the construction of cathodes and test vehicles, their selection for cathode current densities and life burning performance.

An analysis of the life burning results is made. Conclusions and recommendations for future study are made from these finding.

2.0 PROGRAM OBJECTIVES

The objectives of this program of thermionic cathode study was to evaluate the life capabilities of those different electron tube cathode types under high cathode current densities with the lowest possible cathode temperatures under dc voltage conditions.

The three selected cathode types, barium-strontium oxide, coated particle and pore type dispenser, were selected for life burning according to Table 1, Electrical Test Procedures (JPL Specification).

This table divides the selected diodes (48 total) into three groups of 16 diodes for each cathode. Each group of diodes containing its own individual type of cathode is divided into four sub-groups numbered T_i, T_2, T_3 and T_4.

The division of each sub-group was determined by a zero field plot (Schottky plot)[1] as shown in Figure 1. The diodes were measured at cathode temperatures high enough to give zero field extrapolations of current density to meet the desired levels as indicated in Table 1.

Each of the four diodes that complied with the required zero field current densities were burned on life at the pre-determined cathode temperature at two different current densities (2 diodes - each test condition) which were equal to or below the zero field current density. The diodes were measured for zero field emission under dc voltage conditions. The selection of cathode current operating conditions should be low enough to insure space charge operation of the diodes during the life burning cycle of the vehicle.

Because of the small sample size of each unit, the current density was varied from a low to a high level at four different temperatures in the hopes of establishing a pattern of life expectancy and cathode behavior for each type of thermionic emitter.

After selection according to the specifications (Table 1) the diodes were burned on life at the predetermined cathode temperature at a constant dc voltage at the specified cathode current loading. This anode voltage and cathode temperature were held constant for each individual diode throughout the life burning cycle to show the changes as they occur in cathode current with constant cathode temperature.

At each interval of test during life burning, the diodes were measured for cathode current at the specified voltage and cathode temperature. The current at ± 20% of the anode voltage was also recorded.

At each test interval, the activation status of the cathode was determined by using Bell Telephone Laboratory's "dip test" method.[2] The current at 95% of the operating temperature for the sub-groups was also determined from the "dip test".

The end point of life expectancy for each cathode type was arbitrarily chosen to be 50% of the starting current level for each test unit of each cathode type.

To establish a more meaningful end point would depend on the view point or objectives of the individual researcher working with thermionic cathodes. This end point of life expectancy of the cathode would depend upon the application of the thermionic emitter in an electron device.

The objective of this study is to provide sufficient guide lines for cathode performance and life expectancy as they apply to electron tubes and to help in the selection of thermionic cathodes for use in electron devices.

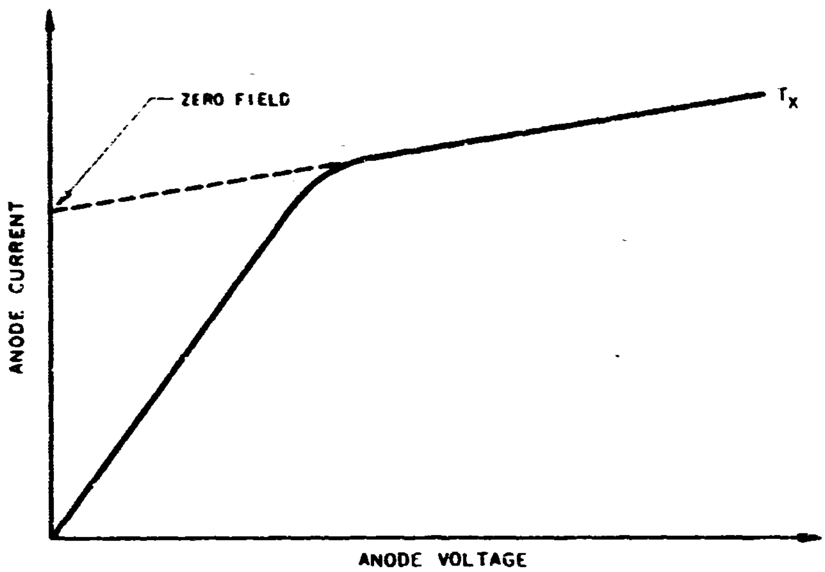

Figure 1 Current Density Level (JPL Specification)

TABLE 1

Electrical Test Procedures (JPL Specification)

	Diode Selection			Life-Test Operation			
	Req'd Units	Life Test Temp	Zero Field Current Dens ma/sq cm	Req'd Units	Current Density ma/sq cm	Req'd Units	Current Density ma/sq cm
Oxide Cathodes	4	T_1	250	2	75	2	150
	4	T_2	500	2	150	2	300
	4	T_3	750	2	225	2	450
	4	T_4	1000	2	300	2	600
CP Cathodes	4	T_1	345	2	138	2	275
	4	T_2	690	2	275	2	550
	4	T_3	1035	2	415	2	830
	4	T_4	1380	2	550	2	1100
Dispenser Cathodes	4	T_1	400	2	200	2	400
	4	T_2	800	2	400	2	800
	4	T_3	1200	2	600	2	1200
	4	T_4	1600	2	800	2	1600

3.0 THERMIONIC CATHODES

The three different thermionic cathodes used in this study were designed and manufactured within the Materials and Techniques Group.

3.1 Pore Type Dispenser Cathode

Fifty pore-type dispenser cathodes were built for use in this study. The pore-type dispenser cathode consists of an emission pellet 0.100 ±0.001 inch in diameter and 0.045 ±0.001 inch thick. This emitter is a porous tungsten pellet impregnated with a molten mixture of barium oxide, strontium oxide and aluminum oxide. Its thermionic emission mechanism consists of a monolayer of barium metal on oxygen on the tungsten face of the cathode.

Barium metal is generated by chemical reaction of the mixture of oxides with tungsten metal in the pores of the cathode. Barium is continually being generated and diffuses through the pores of the tungsten body to the surface of the cathode, replenishing the evaporating barium on the surface.

This pore-type dispenser cathode generally operates at 1100°C and has a work function of 1.9 - 2.1 electron volts. The cathode has capabilities of operating up to 50 amperes/cm^2 under vaying conditions of operation, i.e., pulse or dc operation. Emission failure occurs when the tungsten emitting surface is devoid of barium atoms. The emission failure is generally sudden and catastrophic.

The tungsten pellet is machined from a slug of copper impregnated tungsten which has a controlled porosity of 20 - 22%. The tungsten slug is carefully manufactured by controlled hydraulic pressing and sintering of pure tungsten power capable of passing through a 325-mesh sieve. Following the high temperature sintering, the tungsten slug is impregnated with molten OFHC copper at high temperature in a hydrogen atmosphere.

After machining, the copper in the tungsten pellet is removed by vacuum distillation and the porosity of the body is determined from the loss of weight of copper.

The porous tungsten pellet is polished with fine aloxite paper to a diameter of 0.100 ±.001 inch. It is then inserted into a molybdenum sleeve, 0.103 inch inside diameter and 0.230 inch long, and is peened into the shoulder of the sleeve.

The mixture of barium, calcium and aluminum oxides is now loaded on the emitting face of the cathode and is melted and absorbed into the pores of the porous tungsten body by high temperature heating in dry hydrogen.

After impregnation, the cathode is cleaned by polishing off the excess mixture. The cathodes are stored in vacuum until used.

3.2 Standard Barium-Strontium Oxide Cathode

The standard barium-strontium oxide cathode (better known as the oxide cathode) consists of a metallic body, machined from 0.1% zirconium metal in pure nickel, upon whose face is sprayed a layer of barium and strontium carbonates.

The metallic body of the cathode consists of a 0.1% Zr-Ni sleeve with an inside diameter of 0.093 ±.001 inch and an outside diameter of 0.C124 ±.001 inch. One end of the sleeve is closed, thus forming the emitting surface of the cathode (thickness = 0.045 inch).

The oxide coating used is a standard Raytheon mixture known as C51-3, which consists of a weighted mixture of Baker's RM No. 3, nitrocellulose, butyl acetate and butyl alcohol. Baker's RM No. 3 is a standard 50 - 50 molar ratio of barium and strontium carbonates which has been used by the vacuum tube industry for over 25 years.

This mixture is sprayed upon the face of the cathode to give a thickness of 0.002 to 0.0025 inch and a coating density of one gram/cm^3.

Approximately 100 cathode bodies were machined to dimension and chemically cleaned by existing Raytheon Company standards. Fifty of these cathodes were sprayed with the standard oxide mixture and were stored in vacuum until used.

The oxide cathode normally operates in the temperature range of 750°C - 900°C and has a work function of 1.0 - 1.2 electron volts. The cathode is generally used for cathode loadings from 0.03 - 0.20 amperes/cm^2 under dc operating conditions.

3.3 Coated Particle Cathode

The third cathode involved in this study is the coated particle cathode as described in the Bell System Technical Journal, December 1967, pp 2375 - 2404.

The cathode at present is being manufactured by Raytheon Company and is being tested and used in several Raytheon vacuum tube devices.

The cathode coating differs from the conventional barium-strontium carbonate coating in that each particle is covered with a thin layer of mond nickel. Total weight of the nickel amounts to 1% ± 0.3% of the total weight of coating.

A fluid-bed process is used to coat the alkaline earth carbonate particles. A mixture of nickel carbonyl and hydrogen is bubbled through a suspension of the particles in amyl acetate at a temperature of 120°C. Nickel carbonyl decomposes to nickel and carbon monoxide, with the nickel deposited on the individual particles of the coating mixture.

When the coating process has been completed, nitrocellulose is added to the admixture of coated particles and amyl acetate to make a spray mixture.

50 cathodes made from 0.1% Zr-Ni were sprayed with the coated particle coating to the same specifications as mentioned in Section 2.2 (T = 0.002 - 0.0025 inch, D = 1 gm/cm^3).

The coated particle cathode operates in the range of 750°C - 850°C and has a work function of 1.2 - 1.4 electron volts. It is claimed that this cathode will operate up to 1 amp/cm^2 for long periods of time.

Theoretically, the system should withstand higher current density loading because of the continuous nickel path through the body of the coating which provides higher electrical conductivity.

4.0 THE DIODE TEST VEHICLE

4.1 Description

The diode test vehicle employed in this study is an adaptation of a quality control vehicle presently used for cathode material testing by the Materials and Techniques Group.

Construction of the diode is shown in Figure 2.

The diode is a plane-parallel type with a large, flat, winged anode, 1 inch square, and a small disc-type cathode which is the top surface of a hollow cylinder.

The diode is constructed of metals compatible for each type of cathode. Both the stem and the glass envelope are made from Corning No. 7072 nonex type hard glass. The dimensions of the sealed-in vehicle are 1.5 inch diameter by 4.25 inch height. The sealed-in glass vehicle is mounted in an octal bakelite base of 1.3 inch diameter.

The cathode is heated to desired temperature by radiation from a heater inserted into the cathode sleeve. Cathode temperature is monitored and controlled by visual observation through a hole in the anode by means of optical or infrared pyrometry. A swing type movable flap is closed over the hole in the anode to limit barium evaporation to the top of the glass envelope during the life burning cycle of the diode. The anode is of heavy construction to lessen overheating of the anode during test and life burning.

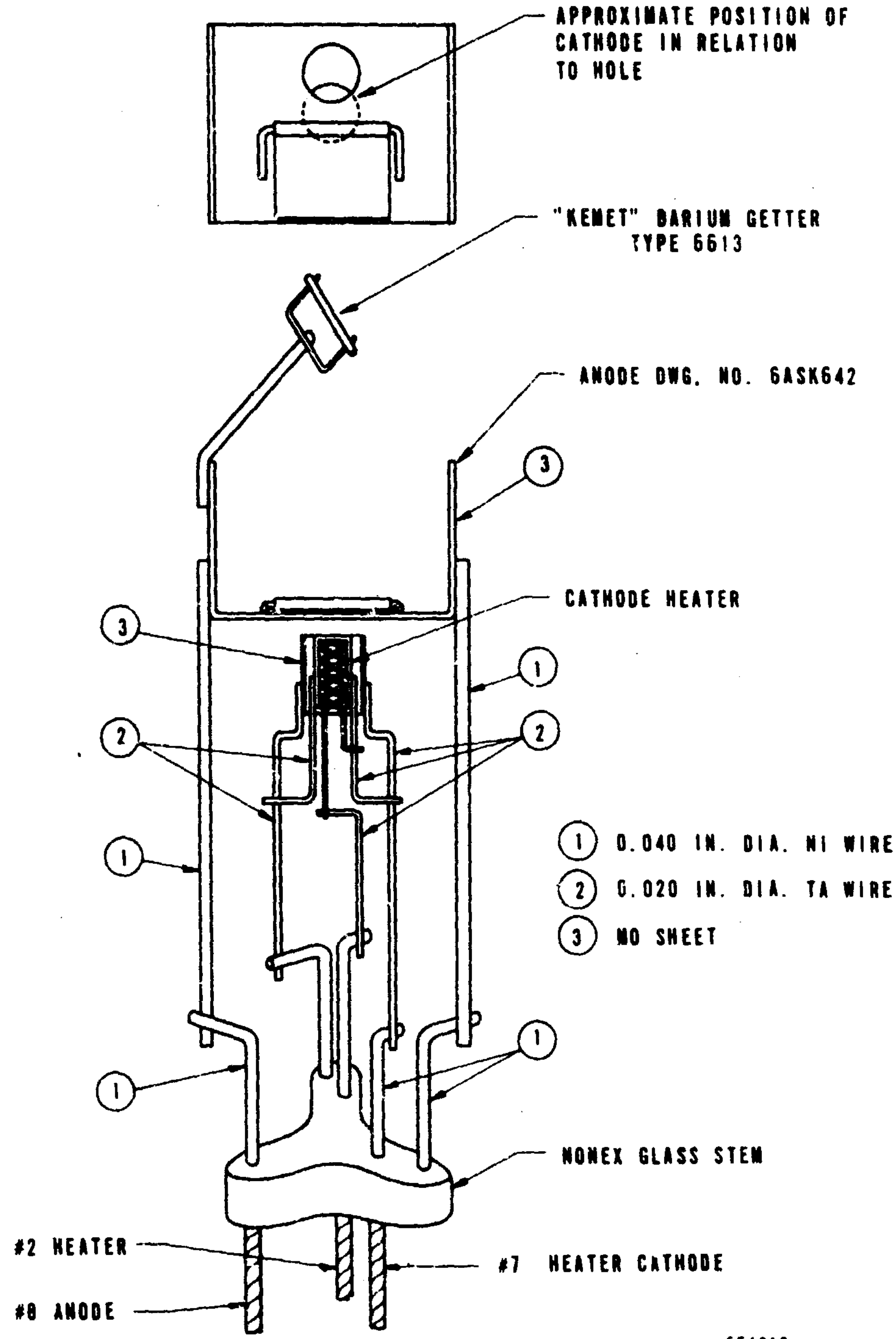

Figure 2. Diode Assembly for Thermionic Cathode Evaluation Study

The maximum dissipation of heat within the vehicle is 8 - 10 watts anode power and 10 watts heater power.

4.2 Construction

A total of 166 diodes were constructed during this period.

Thirty-six diodes using pore type dispenser cathodes were constructed for initial tests (12) and life testing (24). These diodes used the materials shown in Figure 1. All the metal parts were fabricated and chemically cleaned by the cathode section of the Materials and Techniques Group.

Cathode-to-anode spacing was held at 0.015 inch to allow the diodes to operate at a maximum wattage dissipation at 8 - 10 watts on the anode. The most extreme test condition required for the pore type dispenser cathode is E_P = 100 V and i_p = 0.080 mA.

Forty-eight diodes were constructed with the standard barium-strontium oxide cathode, of which 12 were used for initial testing and 36 diodes were used for selection of vehicles for life testing.

The test diodes, built for T_1 and T_2 test conditions were spaced 0.025 inch from cathode to anode. The diodes for T_3 and T_4 test specifications were spaced 0.015 inch from cathode to anode. Forty-eight diodes using coated particle cathodes were constructed for initial test (12), life testing (24) and investigative testing (12). In all cases, the cathode-to-anode spacing was 0.015 inch.

The diodes for the oxide and coated particle cathodes used materials different from those shown in Figure 2. All the metal parts within these diodes were Grade A nickel alloy. The parts were fabricated and chemically cleaned by existing standards by the laboratory.

The construction of the diodes was performed in an air conditioned and humidity controlled room.

The welds in the diode were performed by resistance welding as shown in Figure 3. A stream of helium gas was blown on the weld during the operation to minimize oxidation of the weld joint.

The procedure for chemical cleaning of all the diode parts are included as Appendix 1.

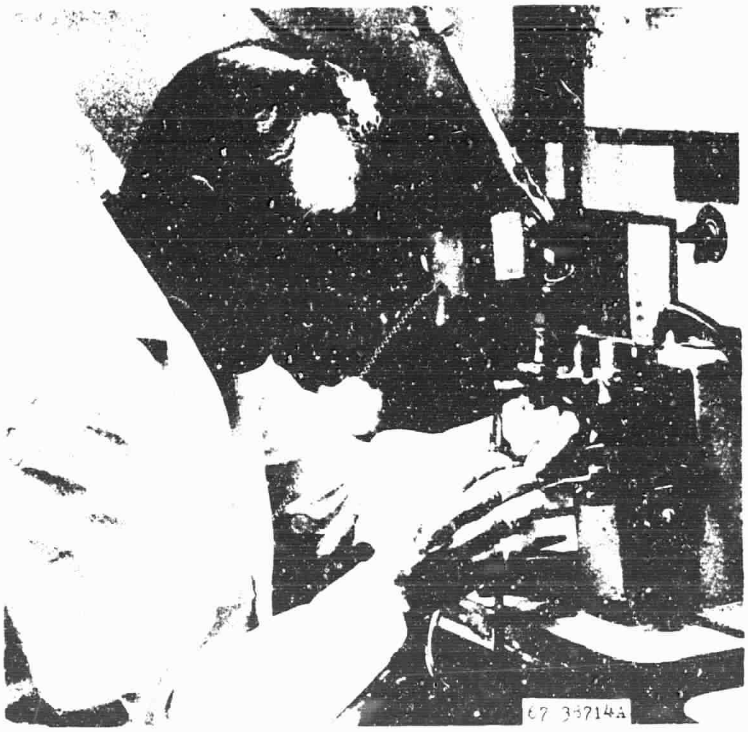

Figure 3. Resistance Welding of Diode Test Vehicle

5.0 EXHAUST PROCEDURES

5.1 Exhaust System

Exhaust processing of all the diode test vehicles was performed on the exhaust system shown in Figure 4. The system is located in a limited access area of the Material and Techniques Group and is under the supervision of the personnel who designed and built the unit originally. The unit has an overall length of 6 feet and width of 3 feet. It is capable of maintaining a vacuum of 10^{-9} torr and is equipped with an electric oven for bakeout of 6 vacuum diodes simultaneously. Each of the 6 exhaust ports has its own 8-litre VacIon pump and vacuum shut-off valve. The system is backed by a K3 Kinney compound pump for rough vacuum pumping and by an oil diffusion pump (200 l/sec) and nitrogen cold trap for medium vacuum range pumping (10^{-3}-10^{-6} torr).

Each glass vehicle is sealed to a glass port which is attached to a stainless-steel, vacuum-tight flange. Each tube has its own vacuum pumping system and cannot contaminate the other tubes during the bakeout and exhaust processing step.

Initial loading, vacuum pumping, and 16-hour bakeout at 450°C are outlined in detail in Table 2.

At this point, the load of tubes should be in the 10^{-9} torr vacuum range and the glass parts should be relatively free of gas and water vapor.

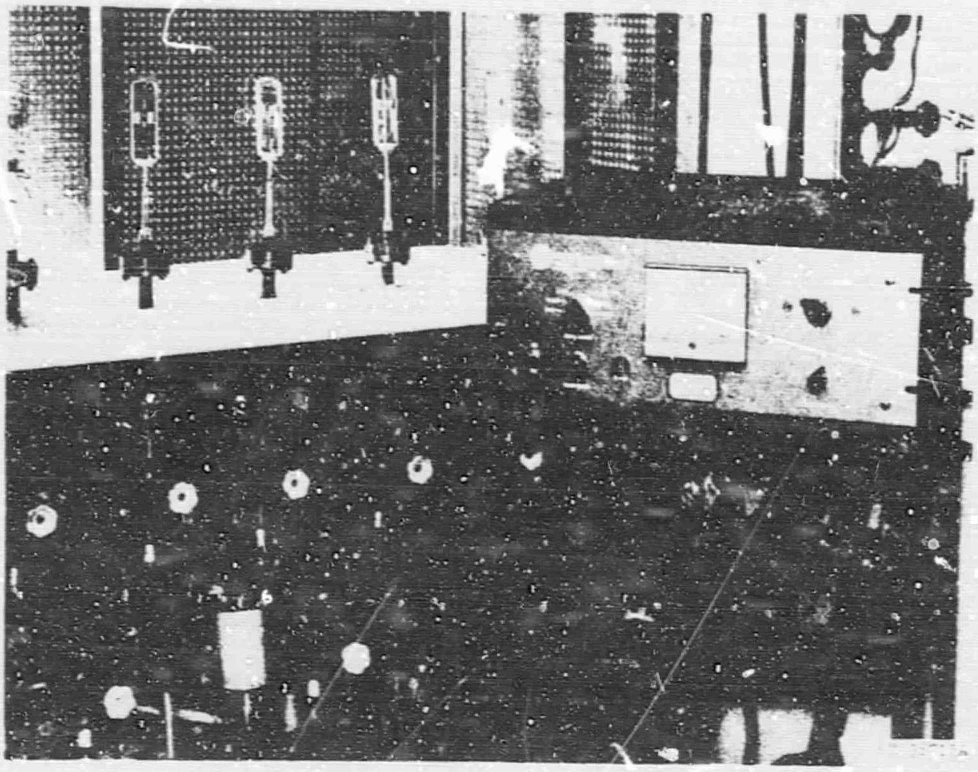

Figure 4 Exhaust System for Diode Test Vehicle (Cathode Laboratory)

TABLE 2

Exhaust Unit Operation Through Bakeout

1.0	Open vacuum valve leading to 6 exhaust positions. (Vac-Ion pumps are off).
2.0	Close valves leading to roughing and oil diffusion pump systems.
3.0	Open bleeder valve, cut off glass exhaust tubes and seal-on 6 glass diodes.
4.0	Close bleeder valve and open vacuum valve to roughing pump. Run until vacuum is at 5 microns pressure.
5.0	Fill liquid nitrogen cold trap, close roughing pump valve and open valve to oil diffusion pump system.
6.0	Pump for 2 hours or until vacuum is less than 5×10^{-6} torr.
7.0	Turn on Vac-Ion pumps and pump until Vac-Ion pumps read less than 5×10^{-5} torr.
8.0	Close off oil diffusion system and valve off each individual Vac-Ion system.
9.0	Pump until the 6 systems are at 1×10^{-6} torr.
10.0	Lower electric oven and heat gradually to 450°C not allowing the vacuum to exceed 5×10^{-5} (2-hour period).
11.0	Bake out diode at 450°C for 16 hours.
12.0	Shut off oven and cool for 2-hour period. Vacuum should be on the 10^{-9} torr range.

5.2 Exhaust Processing of Pore-Type Dispenser Cathodes

The pore-type dispenser cathodes were exhaust-processed according to the schedule shown in Table 3.

TABLE 3

Exhaust Processing of Pore-Type Dispenser Cathodes

a.	After bakeout, the vacuum on each diode should be on the 10^{-9} torr scale.
b.	Slowly heat cathode to 1100°C holding vacuum below 5×10^{-5}.
c.	Hold cathode at 1100°C for 30 minutes.
d.	After 15 minutes of cathode heating at 1100°C, heat anode to 900°C for 1 minute by rf heating.
e.	Degas Getter.
f.	Turn off heater.
g.	Repeat steps b - f for other 5 tubes.
h.	Seal-off 6 diodes. Vacuum on each tube should be on the 10^{-9} torr scale.
i.	Flash Getters.
j.	Attach bakelite base to tubes.

Cathode temperature was read during the exhaust cycle by means of an optical Pyrometer (Leeds Northrup Cat. No. 8622c) aimed through the anode hole.

The main point of interest during the exhaust cycle is the degassing of the cathode and auxiliary metal parts of the tube. Chemical generation of barium metal starts in the cathode pores, and the surface of the cathode shows the presence of barium metal at the tube seal-off step.

In all, 36 diodes were exhaust processed during this study by the aforementioned exhaust schedule. Twelve diodes were used for initial evaluation of the test structure, and 24 diodes were processed for final life test selection. One diode was lost because of an air leak, leaving 23 diodes for life test evaluation.

5.3 Exhaust Processing of the Barium-Strontium Oxide Cathode

Forty-eight test diodes using the standard oxide cathode were exhaust processed during this period. Of these, 12 diodes were used for initial evaluation of the test structure. Twenty-four diodes using 0.015 inch cathode-to-anode spacing were exhaust processed with a yield of 21 acceptable diodes for life testing, and 12 diodes with 0.025 inch cathode-to-anode spacing were acceptable for life test evaluation.

The procedure for exhaust processing is shown in Table 4.

TABLE 4

Exhaust Processing of Barium Strontium Oxide Cathodes

a.	After bakeout, the vacuum on each diode should be on the 10^{-9} torr scale.
b.	Degas Getter.
c.	Outgas anode at 900°C for 1 minute.
d.	Heat cathode to 1050°C keeping pressure below 1×10^{-4} torr (approximately 10 minutes).
e.	Hold 5 minutes at 1050°C.
f.	Drop cathode temperature to 950°C and hold 10 minutes.
g.	Turn off heater.
h.	Repeat steps (b) - (g) for other 5 diodes.
i.	Seal off 6 diodes. Vacuum on each tube should be on the 10^{-9} torr scale.
j.	Flash Getter.
k.	Attach bakelite bases to tubes.

The chemical reactions occurring during the exhaust process of this cathode are entirely different from those of the pore-type dispenser cathode.

In addition to accomplishing the degassing of auxiliary metal parts of the diode, other gaseous end products are produced by the chemical reactions for producing barium metal in the cathode system.

The initial step in cathode processing is the conversion of the cathode coating to the oxides.

$$\text{No. 1 - } \left.\begin{matrix} BaCO_3 \\ SrCO_3 \end{matrix}\right\rangle \rightarrow \left.\begin{matrix} BaO \\ SrO \end{matrix}\right\rangle + 2\ CO_2 \uparrow$$

This is accomplished by heating the cathode as rapidly as possible to an elevated temperature (1050°C) and pumping off the carbon dioxide gas through the vacuum pumping system.

The second step in the processing of the cathode is the generation of barium metal within the oxide coating body.

When the cathode is heated, minute quantities of reducing impurities (i.e., zirconium) will diffuse to the surface of the cathode at the interface of the nickel body and the oxide coating. Zirconium metal will react chemically with either the alkaline earth carbonates or alkaline earth oxide; the reaction is dependant on the state of decomposition of the cathode coating.

If the reducing impurity sees a carbonate, the reaction[4] will be as follows, in the case of barium carbonate:

$$\text{No. 2 - } BaCO_3 + M \longrightarrow BaO + MO + CO \uparrow$$

If the reducing impurity sees an oxide, the reaction will be as follows in the case of barium oxide:

$$\text{No. 3 - } BaO + M \quad BaO + MO + Ba$$

In summarizing the barium generating reactions, the alkaline earth carbonates should be reduced to the oxides as rapidly as possible to prevent the deleterious reactions from taking place (Reaction No. 2).

During the lifetime operation of the oxide cathode, we have a system generating barium continuously. The reducing impurity diffuses to the nickel cathode surface, reacts with barium oxide, produces free barium metal, and eventually evaporates from the coating to the nearest cool surface.

The current loading capabilities of the oxide cathode are dependent upon the number of barium atoms present within the oxide coating. The concentration of barium within the coating will decrease during the lifetime of the tube and will eventually deplete itself. The current capabilities of the oxide cathode will show a gradual decay and eventual failure during the lifetime operation of the cathode.

5.4 Exhaust Processing of the Coated Particle Cathode

Forty-eight test diodes using the coated particle cathode were exhaust processed during this study. Twelve diodes were used for initial evaluation of the test structure. Thirty-six diodes for life test evaluation were exhausted with a yield of 32 diodes for test. The procedure for exhaust processing is shown in Table 5.

Chemical reactions during this exhaust processing are the same as in the case of the barium-strontium oxide cathode. The cathode is processed very slowly to 850°C to convert the alkaline earth carbonate to the oxides and to minimize the oxidation of the nickel particles in the body of the coating. The barium producing mechanism is the same as in the case of the oxide cathode.

Both types of cathode should generate the same amount of barium metal and have approximately the same life capabilities and the same failure mechanism.

The only chemical mechanism present that will reduce the free barium supply is the oxidation of the nickel member of the coating during cathode processing and the subsequent reduction of the nickel oxide by the free barium present in the coating to form barium oxide.

TABLE 5

Exhaust Processing of Coated Particle Cathodes

a.	After bakeout, the vacuum on each diode should be on the 10^{-9} torr scale.
b.	Degas Getter.
c.	Outgas anode at 900°C for 1 minute.
d.	Heat cathode slowly to 850°C keeping pressure below 5×10^{-6} torr (approximately 90 minutes).
e.	Raise cathode temperature to 1050°C rapidly and hold for 20 minutes.
f.	Draw 50 miliamperes of current to anode and hold for 3 minutes.
g.	Drop cathode temperature to 950°C.
h.	Repeat step (f).
i.	Drop cathode temperature to 850°C.
j.	Repeat step (f).
k.	Turn off heater.
l.	Repeat steps (b) - (k) for other 5 diodes.
m.	Seal off 6 diodes. Vacuum on each tube should be on the 10^{-9} torr scale.
n.	Flash Getter.
o.	Attach bakelite bases to tubes.

6.0 ELECTRICAL TESTING AND SELECTION OF TEST DIODES FOR LIFE BURNING

6.1 Electrical Test Procedures

The selection of test diodes was made in accordance with the objectives of the program as outlined in Section 2.0.

The diodes were tested for zero field current levels on the electrical test rack shown in Figure 5.

The diodes were mounted on the rack with the anode flap open. After the cathodes were heated, the temperature of each diode was adjusted to the desired level by means of a 4 ohm variable resistor in each heater circuit. The temperature was monitored by means of an optical Pyrometer (Leeds Northrup Cat. No. 8622c). The anode flap was then closed and the cathode current was recorded from 0 to 150 V dc in 25 V steps. The zero field plot was extrapolated from these data by plotting the cathode current in A/cm^2 against $\sqrt{v}$. The temperature limited current slope of the plot was extrapolated back to the zero voltage point and the intercept of the y axis was taken as the zero field current (Figure 1).

It should be noted that the areas of the dispenser-types cathodes was 0.05 cm^2 and the area of the barium strontium oxide and coated particle cathodes was 0.0785 cm^2.

The activation status of the cathode was measured by using the Bell Telephone Laboratory's "dip test" method.[3,4]

The test circuit for the diode is shown in Figure 6 for dip testing of cathode activity.

The diode was set into the socket with the anode flap in the open position. Then, the infrared pyrometer was pointed at the cathode surface

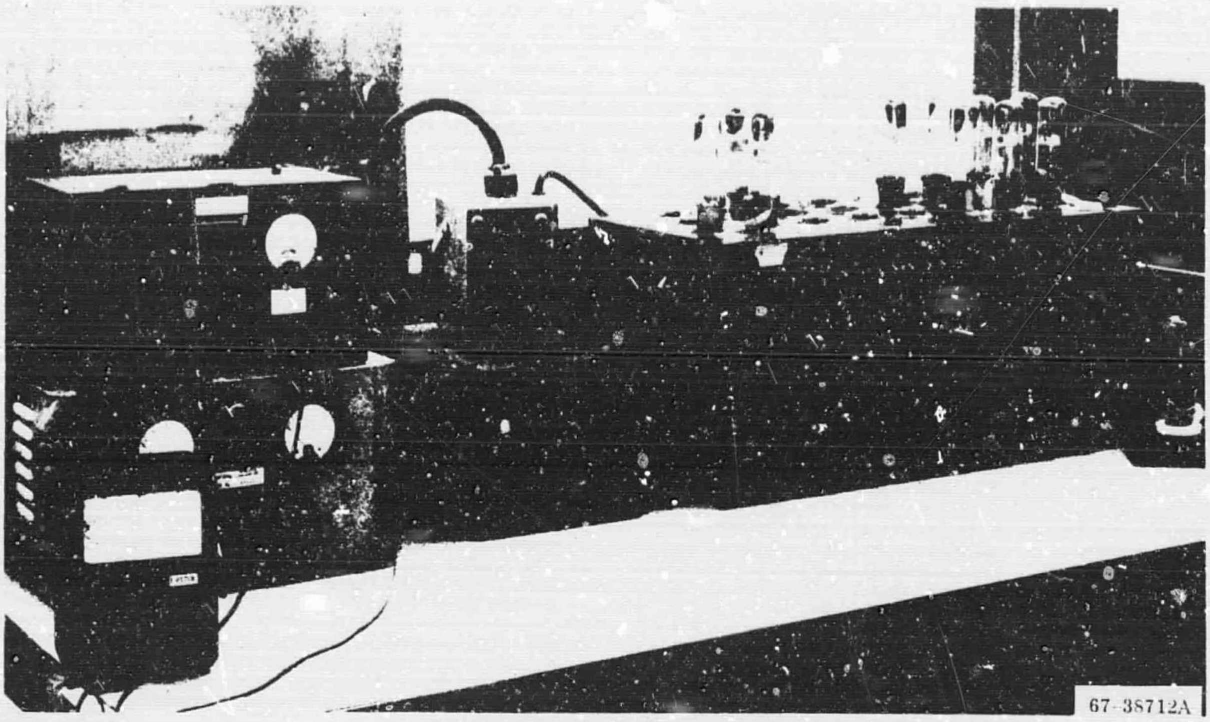

Figure 5 Diode Electrical Test Rack (Cathode Laboratory)

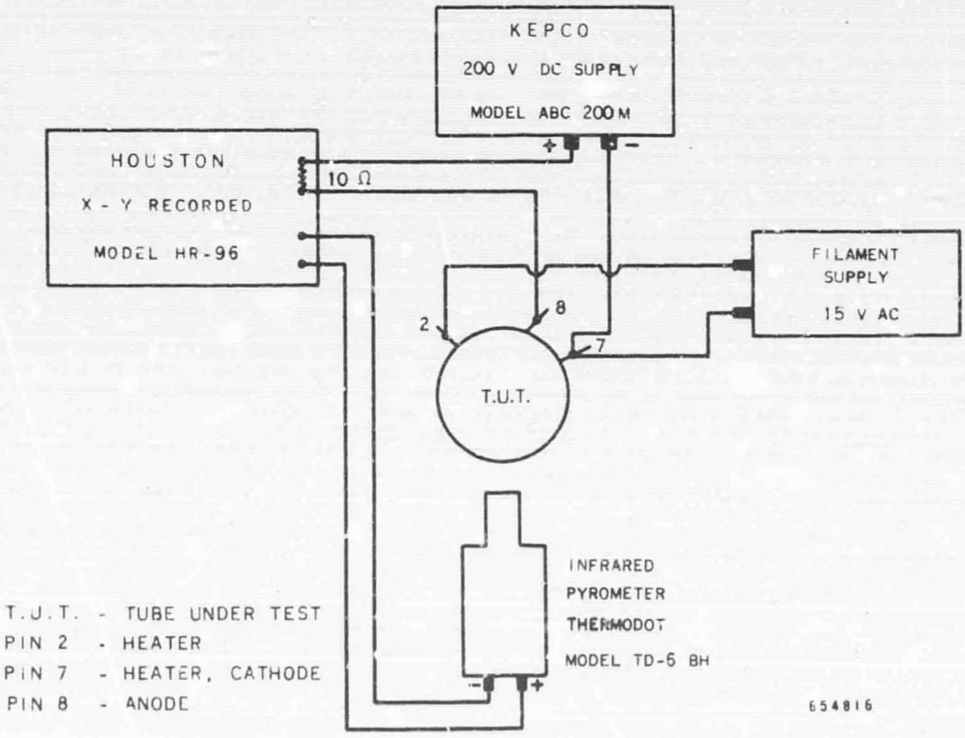

Figure 6 Dip Test of Diode Vehicles

and was calibrated and set to read the optical temperature reading of the cathode from the prescribed temperature to room temperature on the X-axis of the recorder. The anode current was set on the Y-axis to read from the desired current level to 0 current.

After the diode was properly set for cathode brightness temperature and anode current, the heater voltage was turned off to allow the changes in characteristics to be recorded. When the anode current reached 50% of its initial level, the heater voltage was turned on again. The characteristic curve obtained by this method was used to determine the dip temperature. The temperature-limited portion of the curve was extrapolated back to the initial current level on the Y-axis, where the point of intercept on the X-axis was taken as the dip temperature.

Diodes which successfully achieved the zero field current levels and showed sufficient dip temperature were used for life test evaluation of the three cathode types.

6.2 Pore Type Dispenser Cathodes

The test diodes using pore-type dispenser cathodes were numbered for identification: M1 through M24.

Two diodes were first tested for zero field current density using pore type dispenser cathodes. The current level for tube M-3 and M-4 is shown in Figure 7.

For these two diodes, 900°C was determined to be the proper temperature for operation of the diodes under the T_1 condition for pore-type dispenser cathodes (Table 1, Electrical Test Procedure). The 2 tubes had a zero field current level of 0.4/cm^2 at 900°C (0.020 A x 20 = 0.2 A/cm^2). However, a subsequent dip test showed the two tubes to be operating in the temperature-limited emission region.

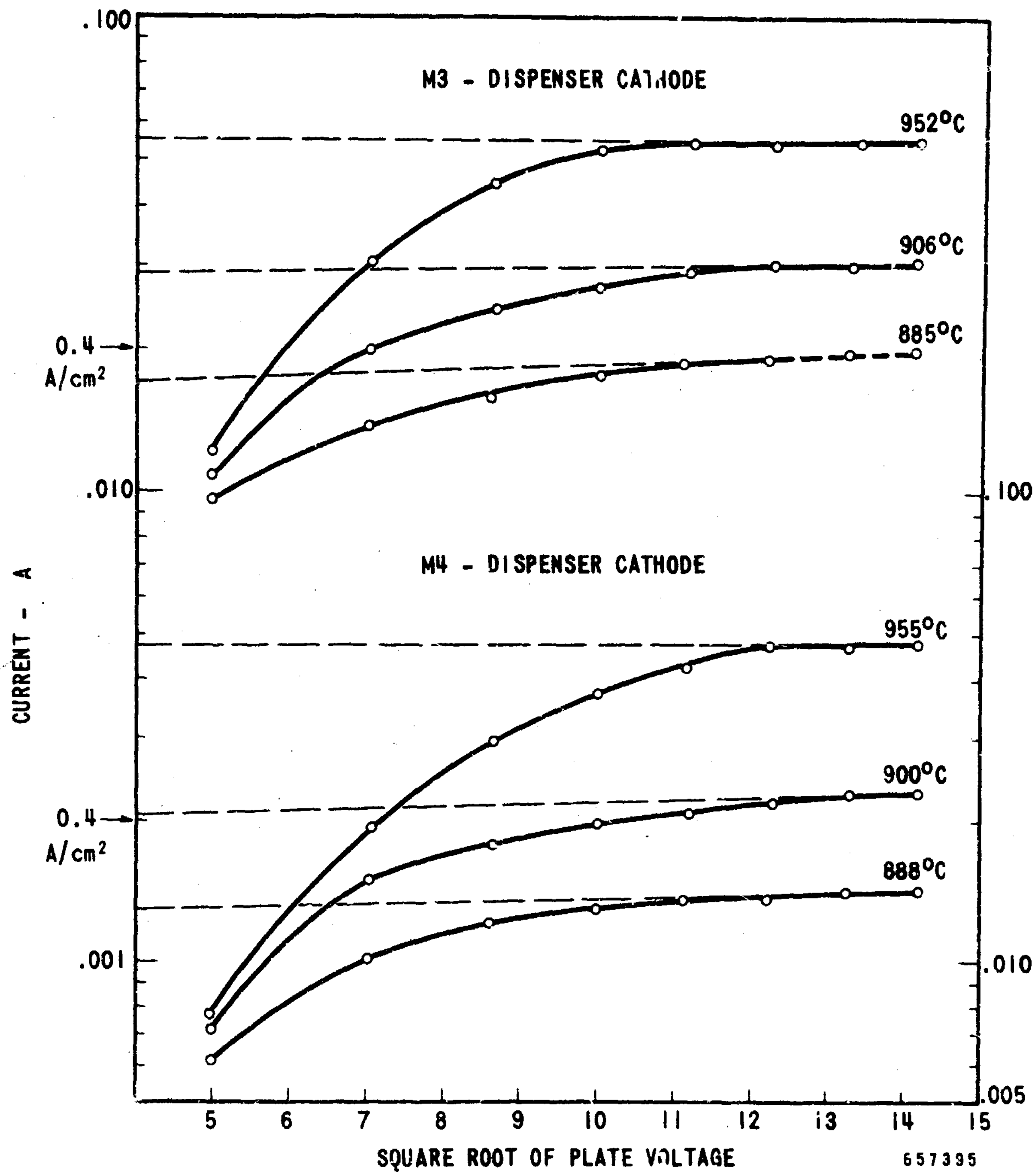

Figure 7. Zero Field Current Density

It was also found that the prescribed zero field current levels for T_2, T_3 and T_4 diode operation were too low for pore-type dispenser cathodes. The diodes were all temperature-limited under these conditions and showed no drop in dip temperature from the operating temperature.

After a conference with the JPL representative, it was decided to raise the zero field current levels at each test condition to give a dip temperature approximately 50°C lower than the operating temperature. Table VI shows the selected zero field current levels and temperatures used for test and evaluation of pore-type dispenser cathodes.

The tubes tested at T_4 did not show current saturation at 1100°C at the highest anode voltage (200V) that could be used on the diode. Above this voltage, the diodes showed overheating of the anode with the cathode current running far above normal current ratings. It is estimated that at 1100°C the zero field current should be in the range of 2.5 amp/cm^2.

Final temperatures selected were 950°C at T_1, 985°C at T_2, 1035°C at T_3 and 1100°C at T_4.

The dip temperatures were determined for each tube at the four T_1 temperatures and eight operating current levels. The results for the dip temperatures are plotted in Figures 8, 9, 10 and 11.

TABLE 6

Zero Field Current Levels
Pore-Type Dispenser Cathodes

Test	Tube No.	AMPS/cm^2	T °C$_{BR}$
T_1 - 950°C	M 1	0.90	950
	M 4	0.96	949
	M 2	1.00	952
	M 3	1.10	955
T_2 - 985°C	M 7	1.32	988
	M 9	1.22	989
	M11	1.26	988
	M12	1.40	984
T_3 - 1025°C	M13	1.70	1029
	M18	1.60	1025
	M14	1.60	1029
	M17	1.70	1026
T_4 - 1050°C	M21	1.90	1049
	M23	1.[illegible]0	1054
	M19	1.90	1049
	M22	1.80	1053

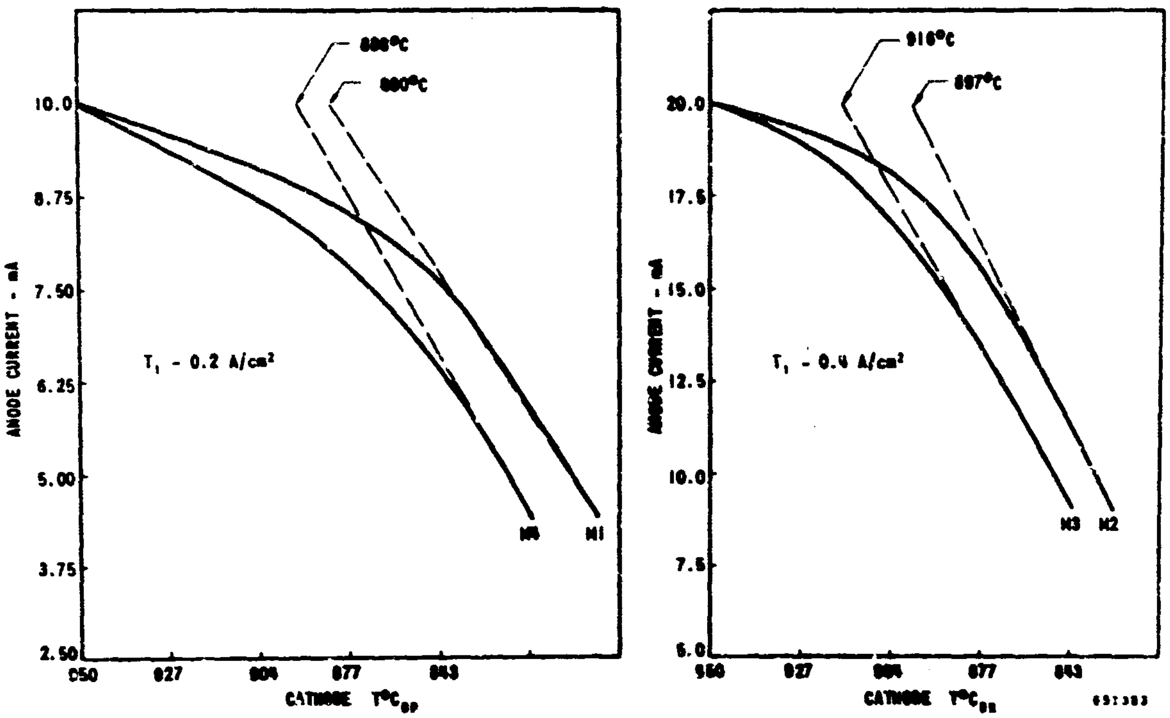

Figure 8 Pore-Type Dispenser Cathode Dip Test

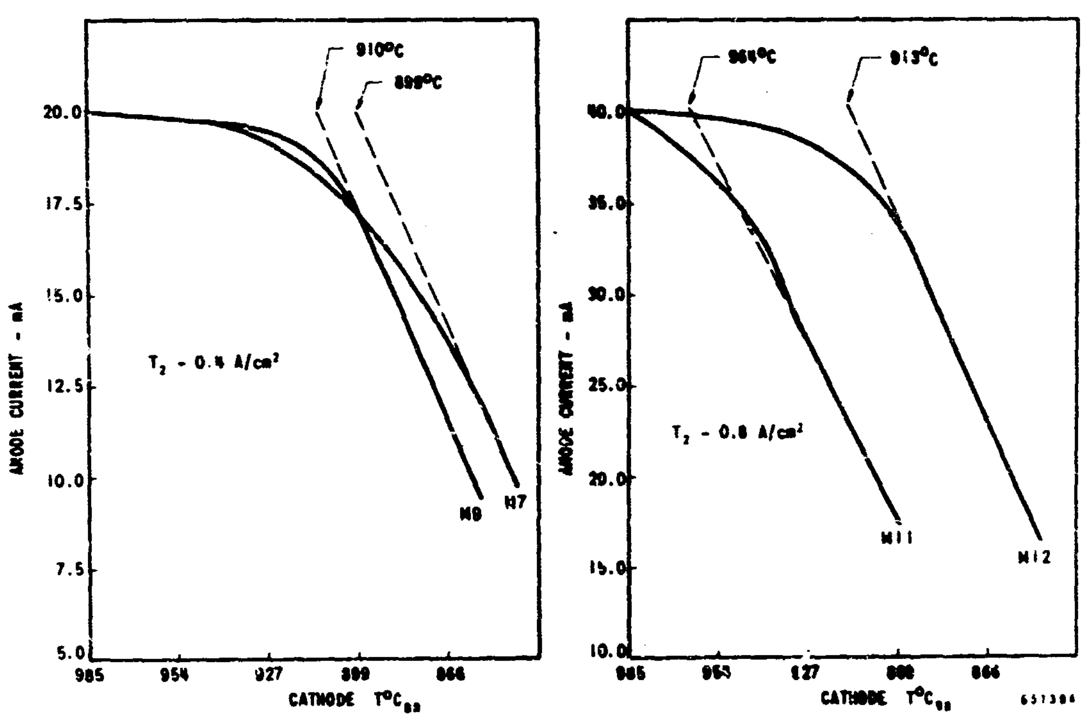

Figure 9 Pore-Type Dispenser Cathode Dip Test

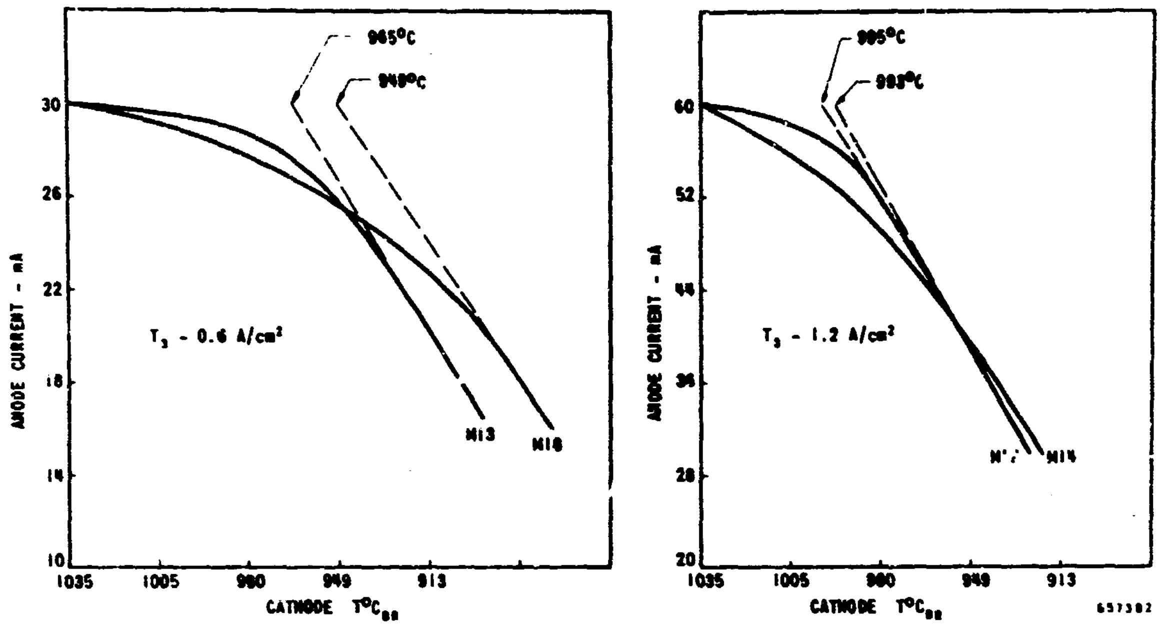

Figure 10 Pore-Type Dispenser Cathode Dip Test

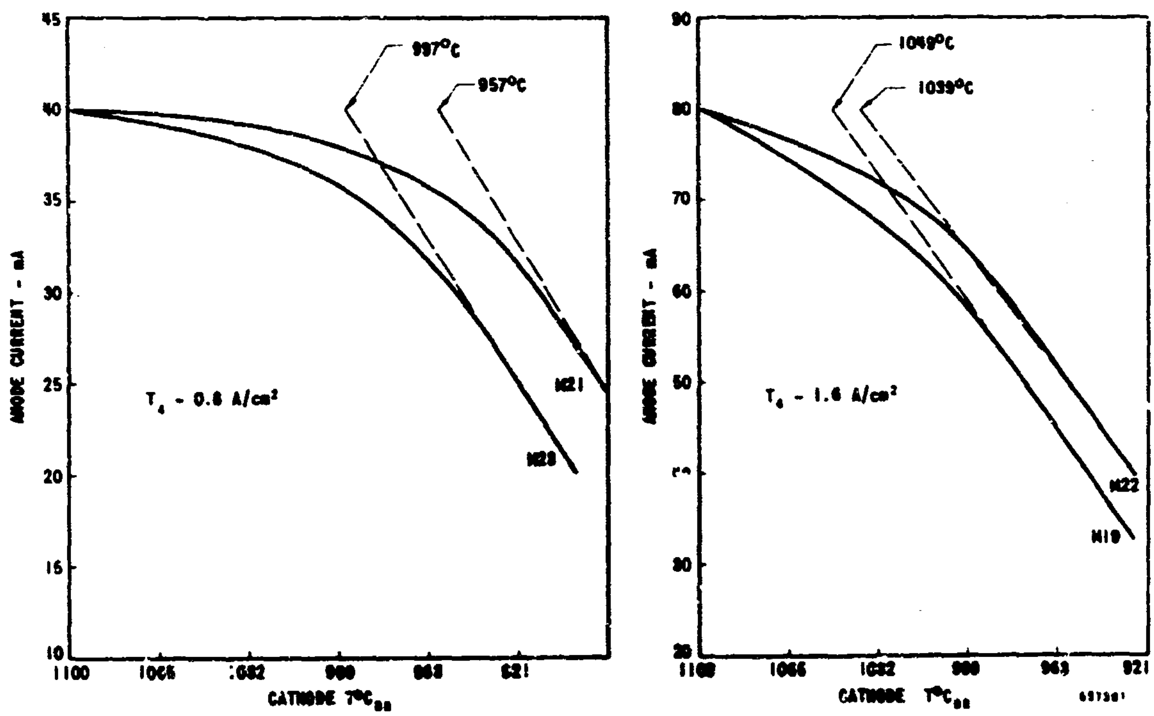

Figure 11 Pore-Type Dispenser Cathode Dip Test

An analysis of the results showed the dip temperatures to be 50 - 103°C lower than the operating temperature at the lower current operating levels with the four different temperature conditions.

At the higher current density levels with the four temperature conditions, the dip temperatures were 21 - 72°C lower than the operating conditions.

At this point, the diodes with the pore-type dispenser cathodes were considered to be ready for life burning.

6.3 Standard Barium-Strontium Oxide Cathodes

The test diodes using the standard oxide cathode with 0.015 inch cathode-to-anode spacing were numbered O1 through O24 for identification.

The diodes were preaged on the electrical test rack (Figure 5) up to 150 hours at 50 volts E_p with the cathode temperature at 850°C.

The diodes started out at very low plate current levels (0 - 5 ms) and gradually built up to the levels shown for a representative sample of 10 diodes in Table 7.

It was also observed that, when testing diodes with oxide cathodes at voltages higher than 100 V dc, the anode current had a runaway tendency toward higher levels. When the diodes showed rapid increases in current levels, a greenish ion glow was observed at the cathode surface. When the voltage was changed to lower levels, the cathode current showed an increase and then a gradual decrease to the levels as shown in Table 7.

It was impossible to obtain good zero-field-emission plots for oxide cathodes because of their failure to saturate at higher dc voltage levels.

TABLE 7

Oxide-Coated Cathodes
Emission Measurements
At 850°C Cathode Temperature

Tube No.	25V	50V	75V	100V
O 1	25	32	37	41
O 2	21	26	26	26
O 4	15	36	50	56
O 7	25	57	98	150
O 8	24	55	92	136
O10	12	31	45	60
O11	15	35	52	62
O12	22	50	61	77
O13	11	25	36	47
O14	10	25	37	50

The estimated zero-field current levels at 850°C cathode temperature are 0.5 A/cm^2 to 0.7 A/cm^2. (Cathode current in mA x 12.7 = A/cm^2.

Because of the difficulty in obtaining satisfactory zero-field emission data, the test diodes were selected by dip testing.

Figure 12 shows the dip-temperature for four diodes selected for operation at the T_4 condition of 0.3 and 0.6 A/cm^2 at 850°C.

Figure 13 shows the dip-temperature for four diodes selected for operation at the T_3 condition of 0.225 A/cm^2 and 0.45 A/cm^2 at 825°C.

The test diodes using the standard oxide cathode with 0.025 inch cathode-to-anode spacing were numbered O30 - O42.

The cathode-to-anode spacing for the diodes needed to meet the low current requirements under T_1 and T_2 conditions had to be increased from 0.015 inch to 0.025 inch because of the unacceptable low plate voltage drop across the diode.

It is desirable to keep the anode voltage above 10 volts dc (at least at 20 volts) to prevent the undesirable 10.0 volt effect.

Eight diodes were selected for life testing under the requirement for oxide cathodes at the T_1 and T_2 conditions.

The dip temperatures for diodes under T_2 conditions are shown in Figure 14 and for T_1 conditions in Figure 15.

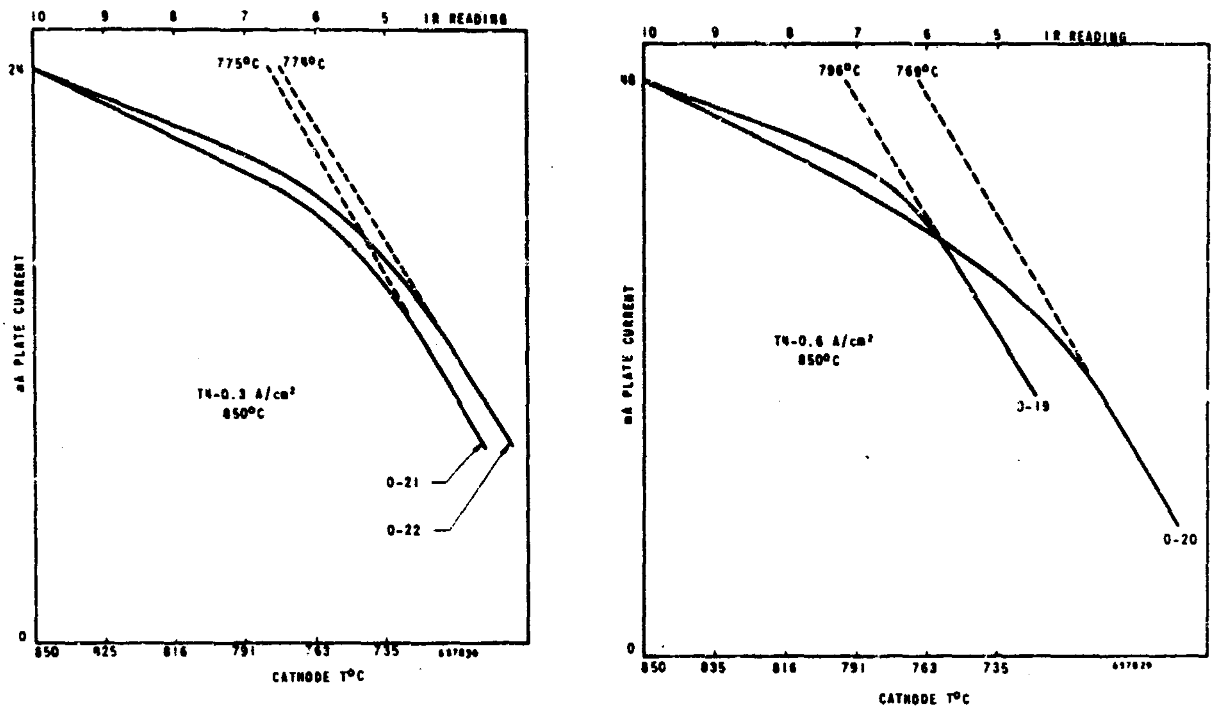

Figure 12 Diodes O-19/20/21/22 - Oxide-Coated Cathode Dip Test

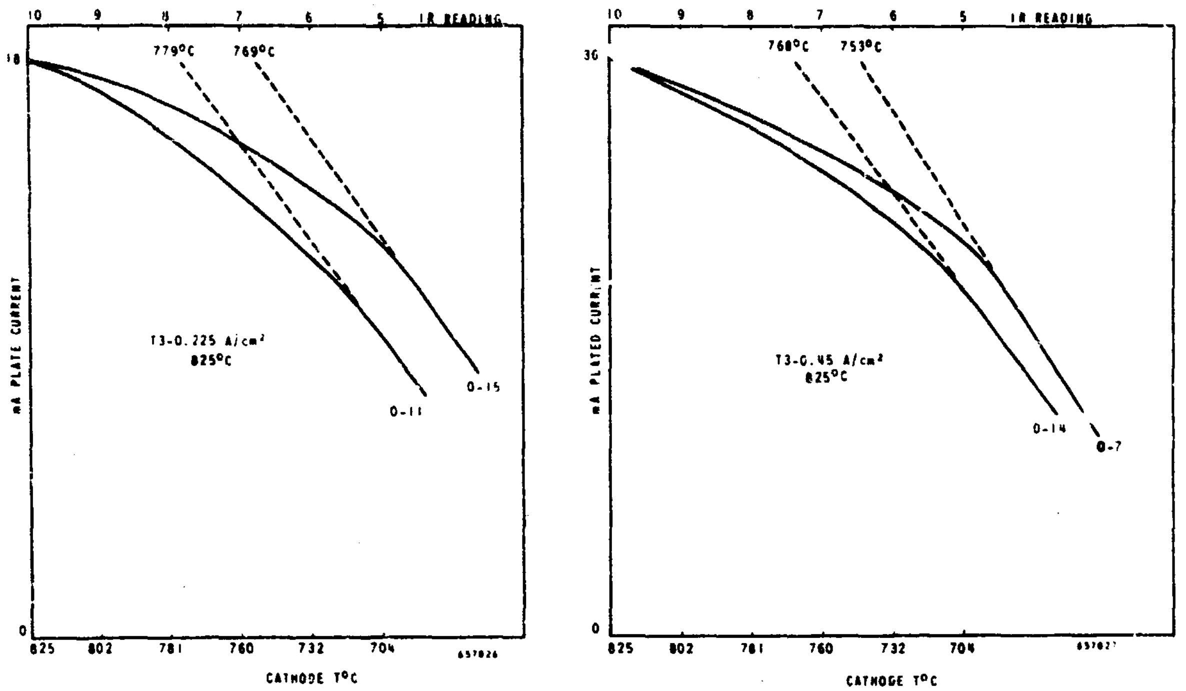

Figure 13 Diodes O-7/11/14/15 - Oxide-Coated Cathode Dip Test

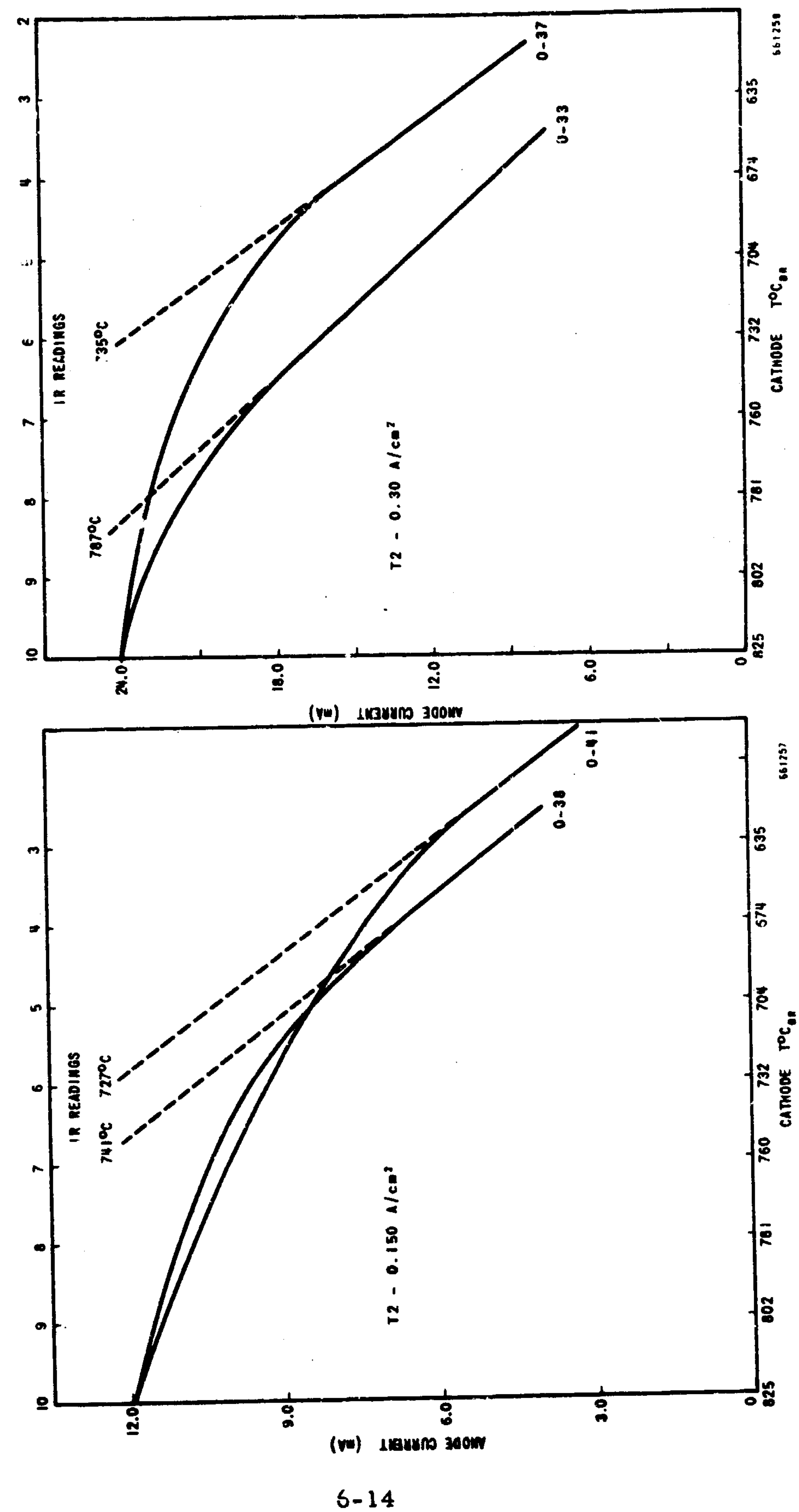

Figure 14 Diodes O-38/41/33/37 - Oxide Coated Cathode Dip Test

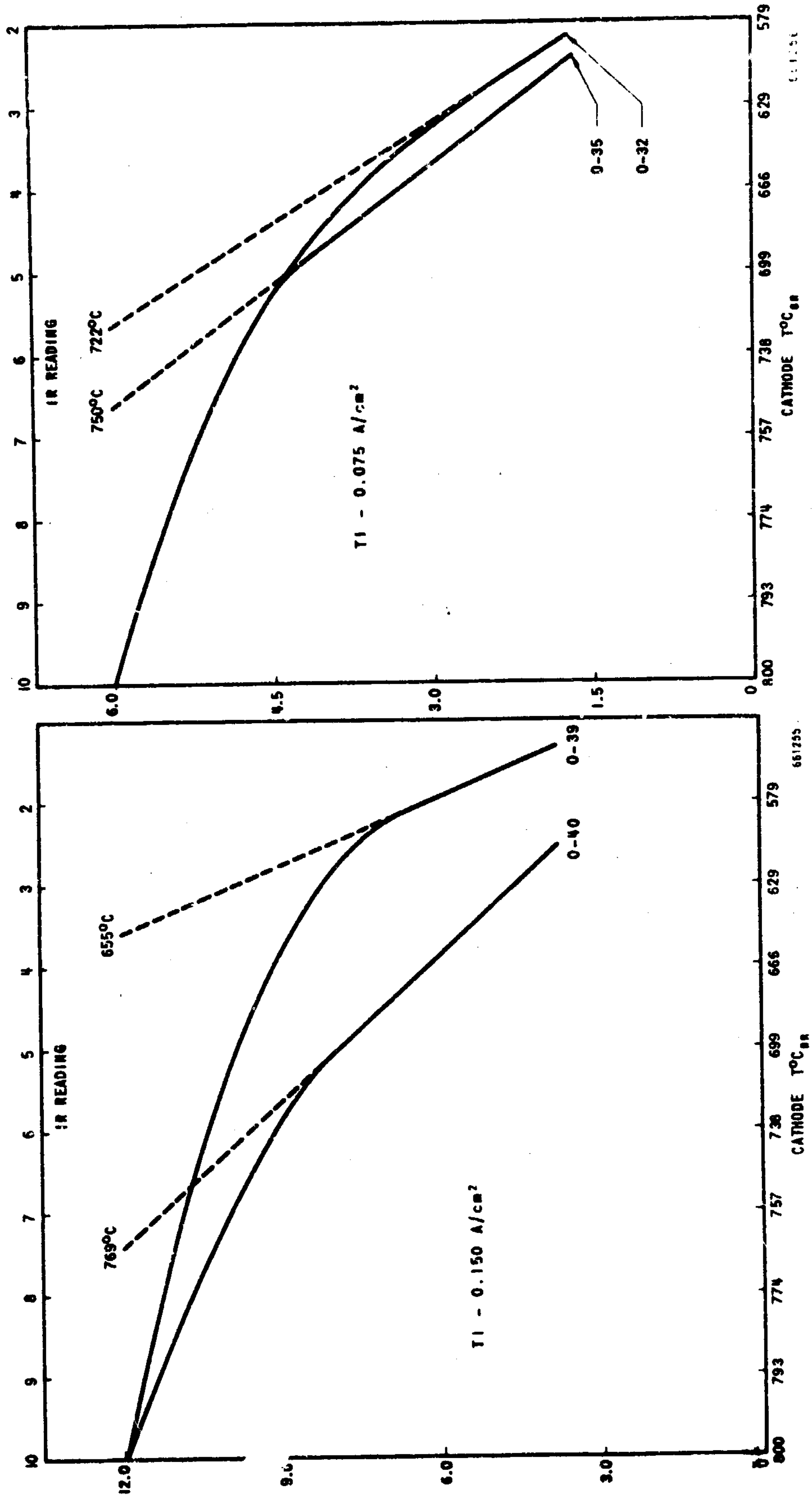

Figure 15 Diodes O-32/35/39/4 - Oxide-Coated Cathode Dip Test

At this point, the diodes using the standard barium-strontium oxide cathode were considered to be ready for life burning.

6.4 Coated Particle Cathodes

The test diodes using coated-particle cathodes were numbered C1 through C24 for identification.

The diodes were preaged on the electrical test rack (Figure 5) up to 150 hours at 50 volts E_p with the cathode temperature at 850°C.

The diodes, at the initial stages of burning, showed very low plate currents (0 - 5 mA) and gradually built up to the levels shown in Table 8 in the forementioned time periods.

Table 8

Coated Particle Cathodes
Emission Measurements
At 850°C Cathode Temperature

Tube No.	25V	50V	75V	100V
C 2	25	53	75	90
C 3	33	39	40	43
C 4	29	41	43	43
C 7	30	57	68	72
C 8	17	37	58	63
C 9	11	27	42	53
C10	24	43	48	50
C14	18	39	54	58
C15	12	27	42	64
C16	20	41	80	gassing

Note: Readings are in milliamperes of anode current.

It was observed, while testing the diodes, that voltages applied to the anode higher than 100 V dc would cause the diode to show runaway current levels.

When the diodes showed rapid increases in current levels, a bluish ion glow was observed at the cathode surface. At this point of testing, when the cathode voltage was dropped to lower levels, the cathode current would be higher than shown in Table 8. If the cathode was left at lower voltage, the current would decay to its original levels very gradually in a period of 10 - 30 minutes.

For this reason, it was impossible to obtain good zero field-emission plots for coated-particle cathodes. Figure 16 shows the zero field-current density plots for diodes C4, C7, at cathode temperatures of approximately 850°C and 800°C. The point to be noted is the small, unexpected change in zero-field current for a change of 50°C in cathode temperature. The estimated zero field-currents at 805°C are ranging from 0.3 to 0.5 A/cm^2 and at 850°C from 0.5 to 0.7 A/cm^2 in measurable diodes.

The diodes with coated-particle cathodes were next tested for dip temperature under T_1 (0.275 A/cm^2) and T_2 (0.55 A/cm^2) operating conditions as specified in Table 1.

The dip-test results for diode no. C-7 are shown in Figure 17. This shows a dip temperature of 770°C for a plate-current level of 0.275 A/cm^2 at 850°C (T_1) and of 877°C for a plate current level of 0.55 A/cm^2 at 900°C (T_2).

Further measurements of other diodes showed the dip temperatures to be between 770 and 830°C at 0.275 A/cm^2 (T_1) and between 870 and 900°C at 0.55 A/cm^2 (T_2).

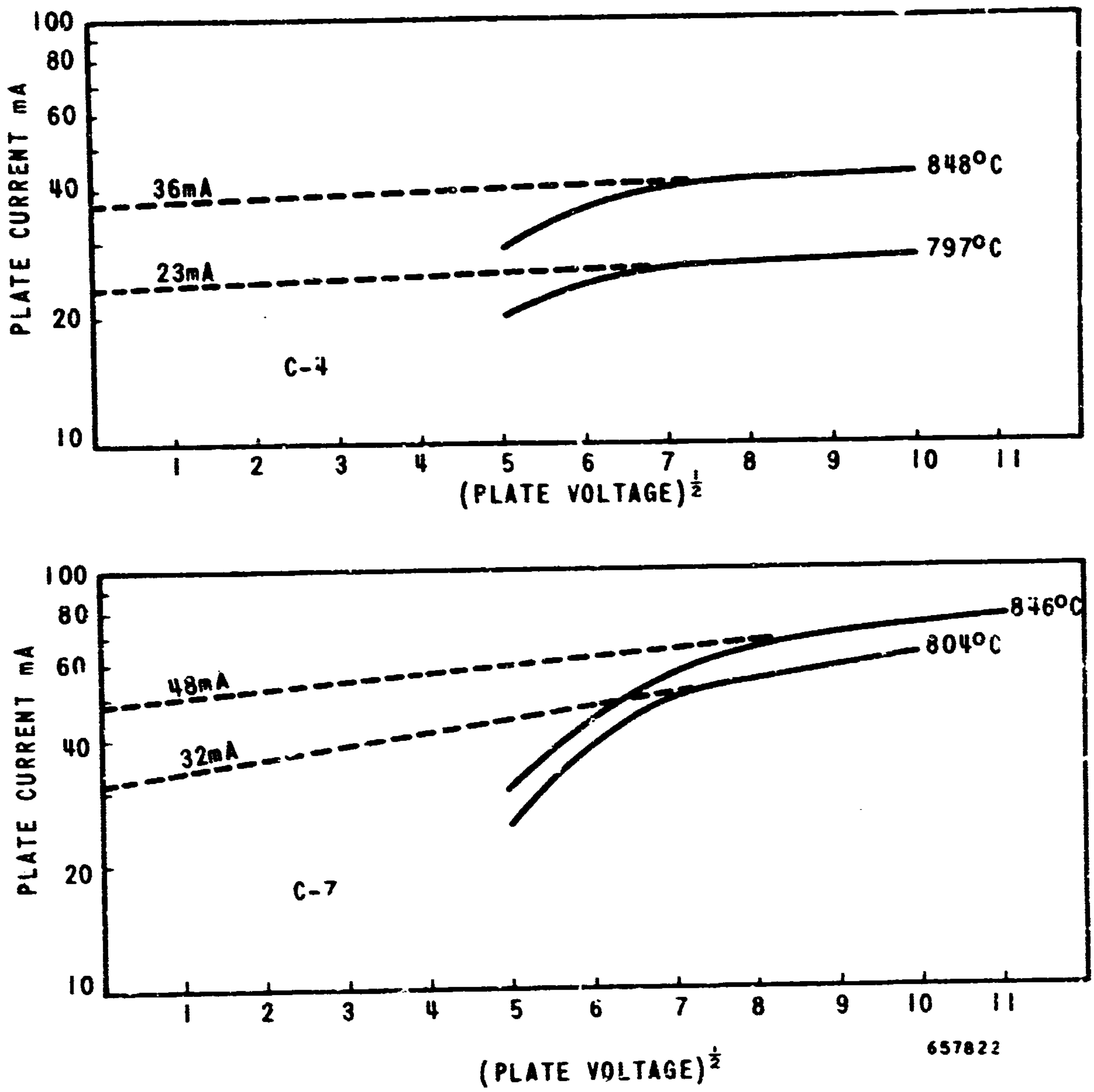

Figure 16 Diodes C-4/7 - Zero Field-Current Density Coated-Particle Cathodes

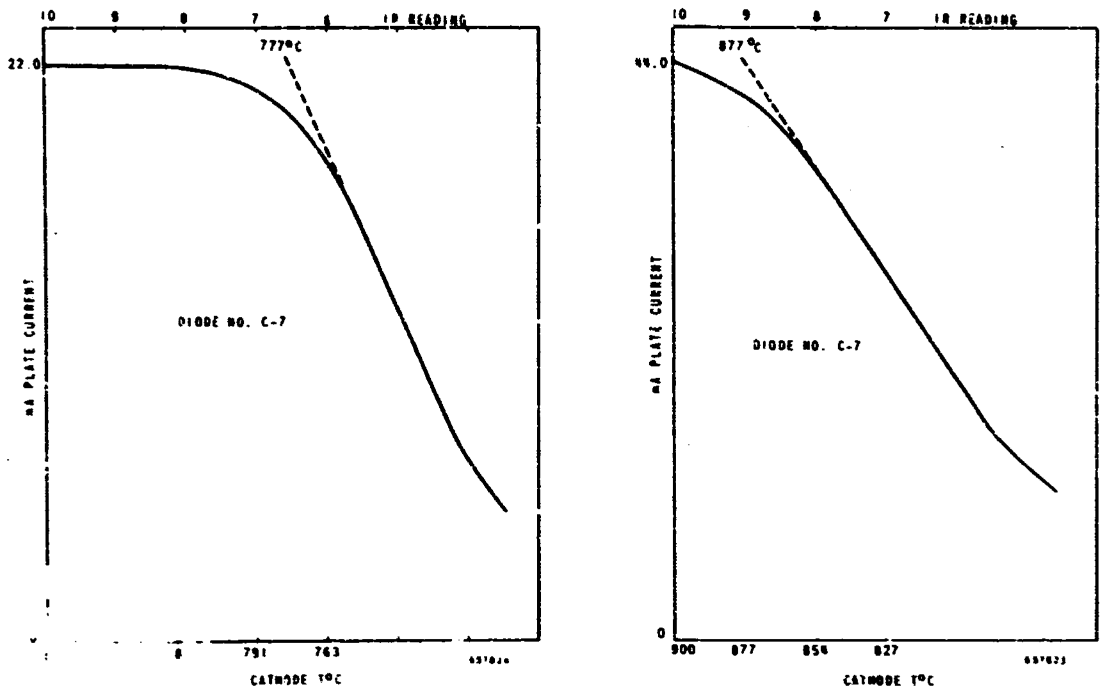

Figure 17 Diode C-7 - Coated-Particle Cathode Dip Test

Dip-temperature measurement at current levels from 0.55 A/cm^2 to 0.83 A/cm^2 at 900°C did not show any space-charge region or measureable dip temperature.

A peculiar change in dip-temperature measurement is noted in Figure 18. Diode No. C-4 was originally measured at 850°C cathode temperature for dip-temperature at 0.275 A/cm^2 and showed the dip-temperature to be 850°C. The cathode temperature was then raised to 900°C and the dip-temperature was determined to be 866°C (Dip No. 1). Then the diode was measured at 0.83 A/cm^2 and the dip-temperature was at 900°C. The test was repeated at 0.275 A/cm^2 and the dip-temperature was lowered to 766°C (Dip No. 2). The diode, on aging 10 minutes at 22 mA, showed a dip-temperature rise to 844°C (Dip No. 3). This condition is repeatable on the same diode and all other diodes at either 850°C or 900°C at 0.275 A/cm^2.

The reason for this change in dip temperature is believed to be caused by temporary electrolytic activation of the cathode.

An analysis of the presented data shows the coated-particle cathode, under the conditions of construction and test, to be capable of meeting the T_1 test conditions (0.275 A/cm^2) at 850°C and the T_2 test conditions (0.55 A/cm^2) at 900°C. Electrical testing at higher current densities at 900°C does not show any appreciable measurement of a dip-temperature.

Because of the high temperatures necessary to maintain the desired current levels (Table No. 1) it was decided not to place the diodes with coated particle cathodes on life burning.

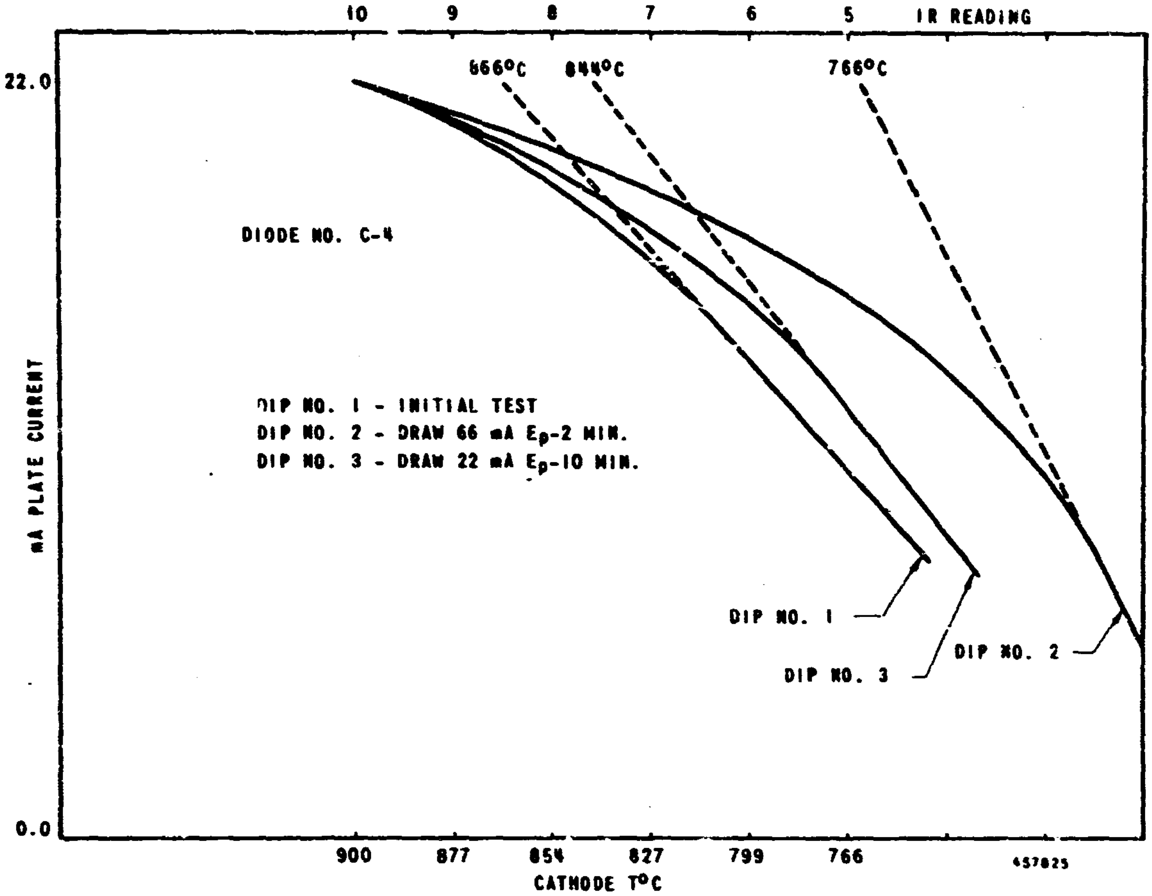

Figure 18 Diode C-4 - Coated-Particle Cathode Dip Test

6.5 Pulse Testing of Oxide and Coated Particle Cathodes

Because of the difficulty experienced with coated-particle and oxide cathodes in obtaining high current densities (1.0 A/cm^2) a series of tests was conducted to compare the dc and the pulsed-voltage behavior of the two cathodes under investigation.

Figure 19 shows the comparison of two oxide cathodes (O-4, 0-12) and two coated-particle cathodes (C-2, C-17) tested under dc and pulsed voltage conditions.

The four diodes under dc operating conditions show deviations from space charge at low current levels.

The same tubes, under pulsed (3% duty cycle, 0.5 ms pulse) show a straight line with a slope equal to 1.23 - 1.30.

An examination of the oscilloscope trace of the square wave form of the pulse does not show any deterioration of the peak pulse line.

It should be noted that the four diodes show the same behavior under pulsed conditions with a deviation from space charge with increased pulse length is not known at this time.

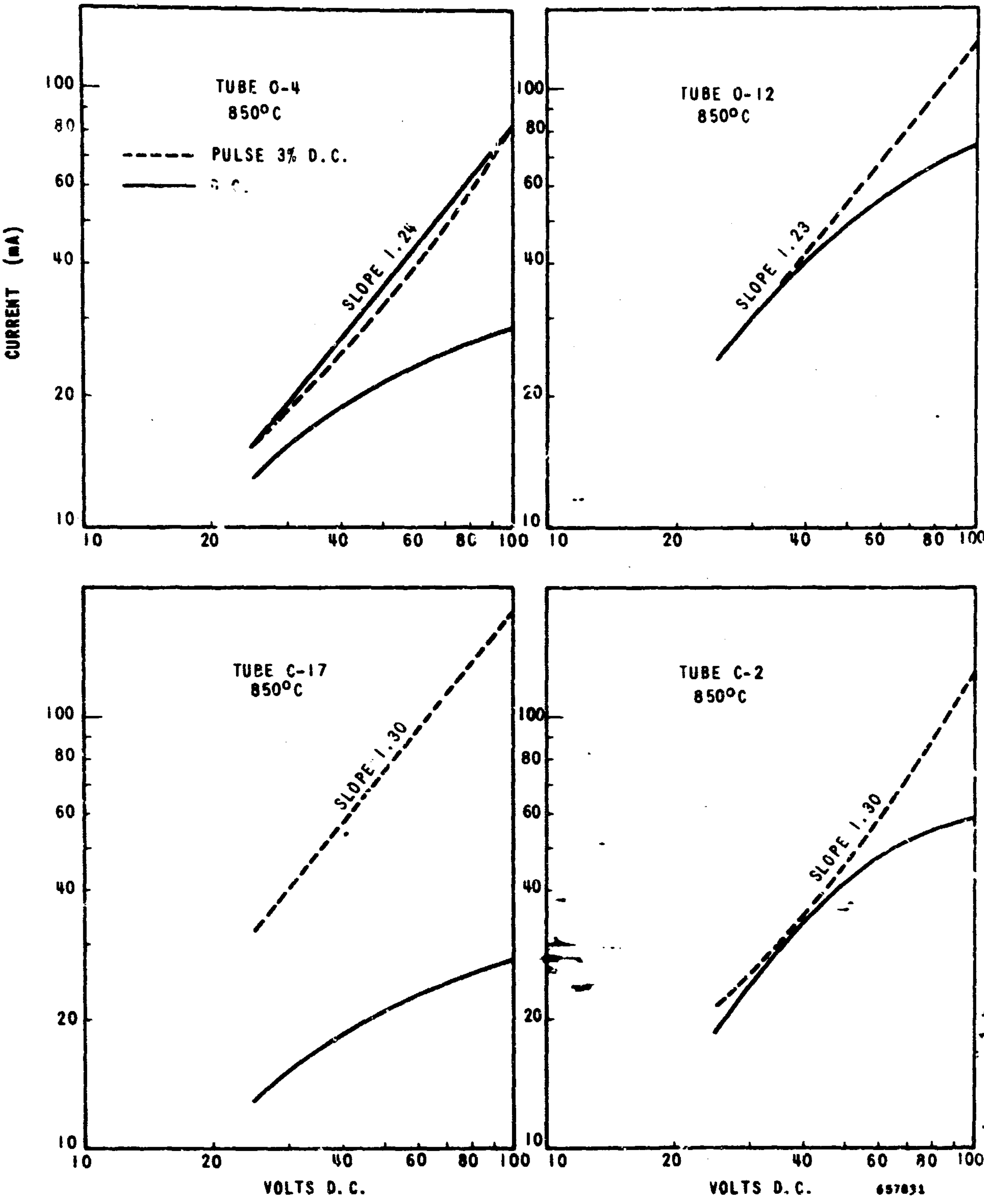

Figure 19 Test Diodes - Dc Conditions Compared to Pulsed Conditions (0.3 ms pulse, 100 pps)

7.0 LIFE BURNING AND TESTING PROCEDURES

7.1 Life Test Equipment

A life test rack was designed by Raytheon personnel and was constructed by the Cober Electronics Company of Stamford, Connecticut for specific use on this program.

The life test equipment was delivered to the Materials and Techniques Group on May 31, 1967 and has been in continuous use until the termination of this contract on September 30, 1969. A photograph of the equipment is shown in Figure 20.

The cathode life test rack, Cober Model No. 1369 has 48 test sockets, with each position having its own controls. The pockets are divided into 3 bands of 16 sockets each. Each bank has its own regulated filament supply which can deliver 24 V ac at 20 A to each bank. Each test position has a 4 ohm, 50 W rheostat to adjust filament current for control of cathode temperature. The cathode temperature can be varied up to 150°C on each bank over the 800°C - 1100°C range setting on the particular bank.

A variable constant plate voltage is supplied to each socket in the range of 20 to 200 V dc. Each socket has its own module for adjustment of constant plate voltage; the modules feed from two Technipower solid state model L-100, 0-6.0 A regulated power supplies.

The constant voltage setting on each socket is monitored by a calibrated 7-1/2 in. square toutband meter, Weston Model 1971. It has a sensitivity of 20,000 ohms per volt with internal multipliers permitting two ranges 0-100 V dc and 0-250 V dc.

The life test rack was installed in a limited access area in the cathode development section of the Materials and Techniques Laboratory.

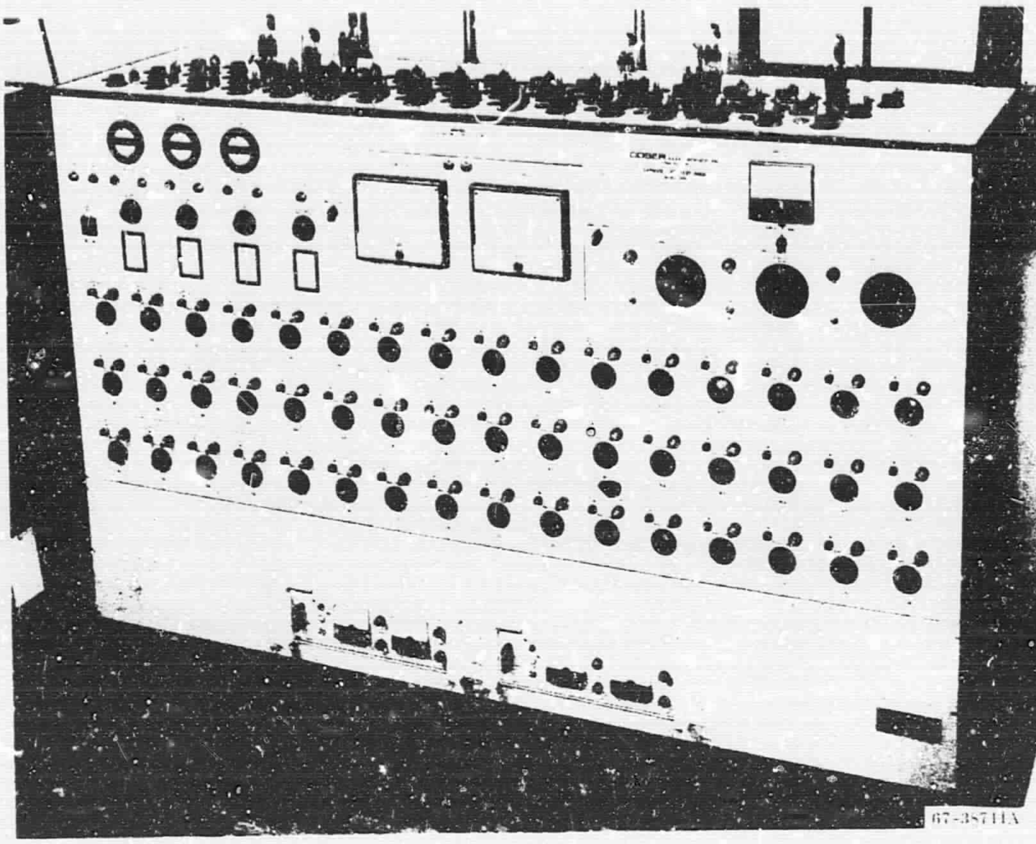

Figure 20 Life Burning Equipment

7.2 Life Test Procedure

The test diodes were placed in groups of four into the same bank of sockets according to the required temperature specifications and cathode type.

The life test rack was turned on and the plate voltage supplies were set at 110 V dc. The filaments were raised to a predetermined voltage and each diode was calibrated for cathode temperature by means of the adjustable 4-ohm rheostates. The temperature was monitored by an optical pyrometer through the open anode flaps.

The anode flaps were then closed and the individual anode voltage modules adjusted to give the constant plate voltage necessary to draw the required cathode current as specified in Table 1.

At each interval of life testing, the cathode current was recorded at the predetermined anode voltage. The cathode current was also read at ±20% of the specified voltage for each test diode.

The diodes were removed from the life test rack at each test period, and were read for dip temperatures according to the procedures outlined in Section 6.1. The cathode current was also determined for 95% of the operating temperature from the dip temperature curve tracing.

The diodes were then replaced on the life test rack and were recalibrated for cathode temperature and anode voltage.

7.3 Pore-Type Dispenser Cathodes

The test diodes with pore-type dispenser cathodes under T1, T2 and T3 conditions were placed on life burning on August 24, 1967 and completed 16,805 hr of life burning as of the termination date of this study.

The test diodes under T4 conditions were placed on life burning on August 31, 1967 and had completed 16,502 hr as of September 30, 1969.

During this time period of twenty-five months of operation (approximately 18,240 hours), the life rack was shut down during two vacation periods in 1968 and 1969 for a total of forty-eight days and for five week-ends (total of ten days). Four week-end shut downs were power shut-downs for utility company service. The other shut-down was for life test rack servicing. This consisted of clean-up of contact points in the plate voltage module switches.

The life burning results for the pore-type dispenser cathodes are shown in Figures 21 (T1), 22 (T2), 23 (T3) and 24 (T4).

The other life test measurements are shown in Tables 9 (T1), 10 (T2), 11 (T3) and 12 (T4).

Three of the four diodes have successfully passed 16,502 hours of life burning under T4 conditions.

Twelve diodes have passed 16,805 hours of life burning under T1, T2, and T3 conditions.

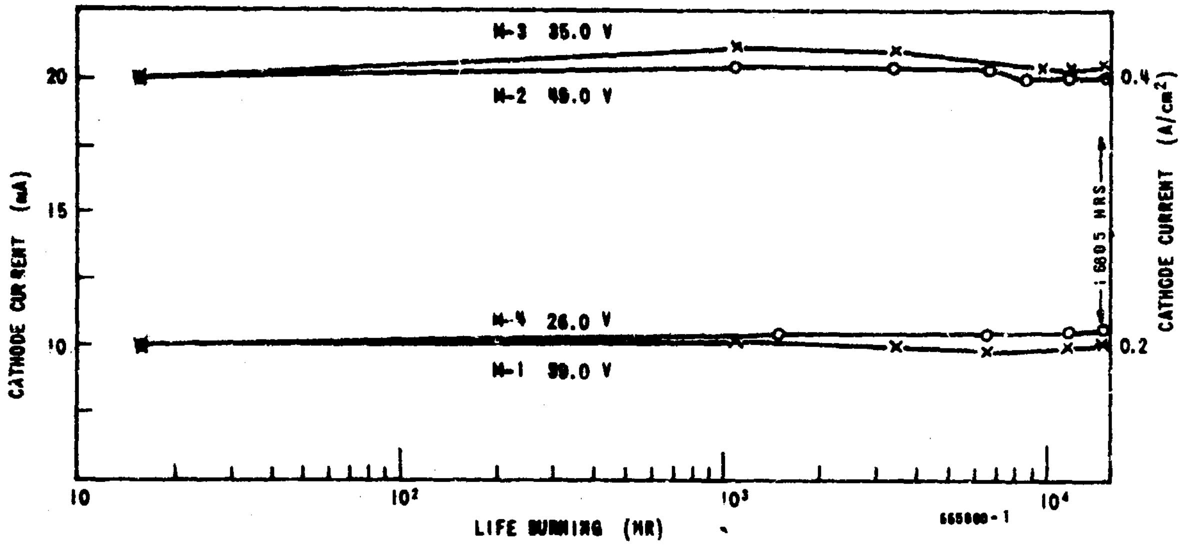

Figure 21. Pore-dispenser Cathode - T1 = 950°C_{BR}

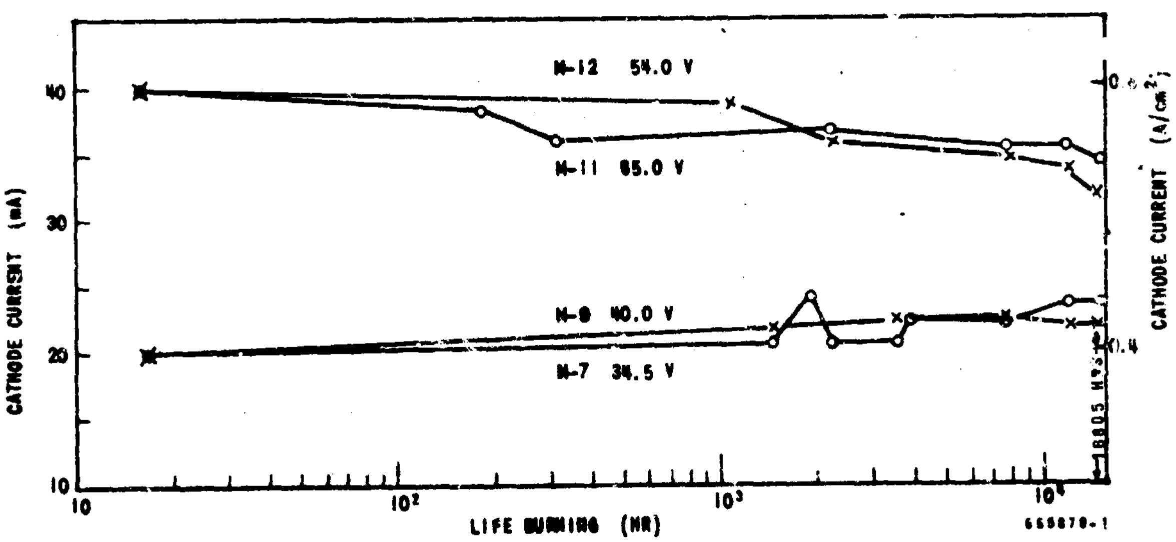

Figure 22. Pore-dispenser Cathode - T2 = 985°C_{BR}

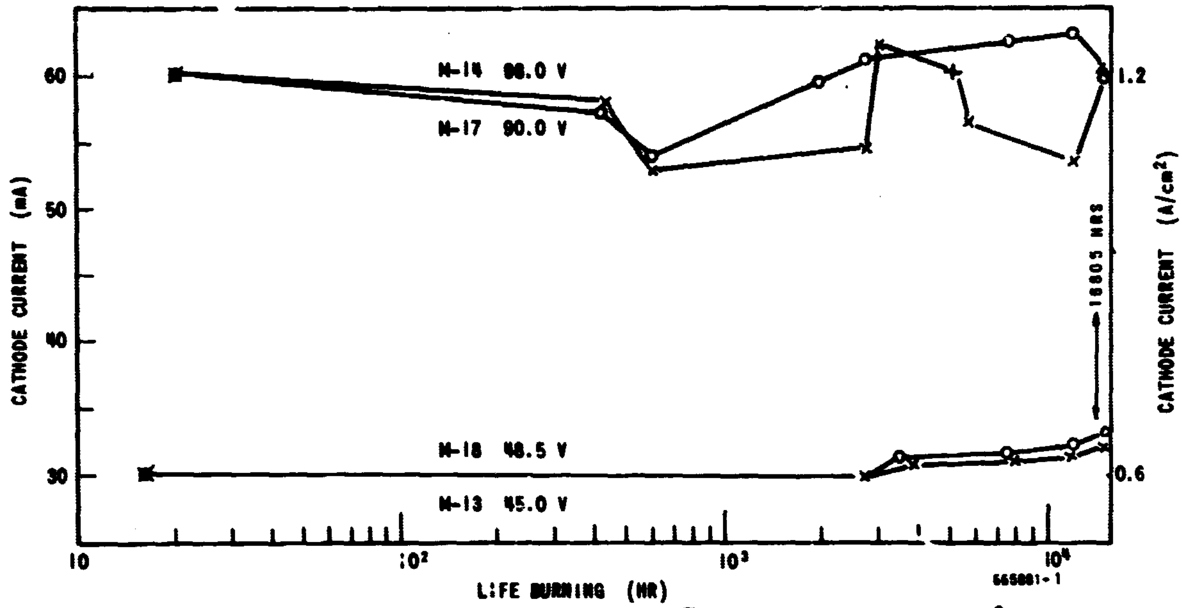

Figure 23. Pore-dispenser Cathode - T3 = 1035° C_{BR}

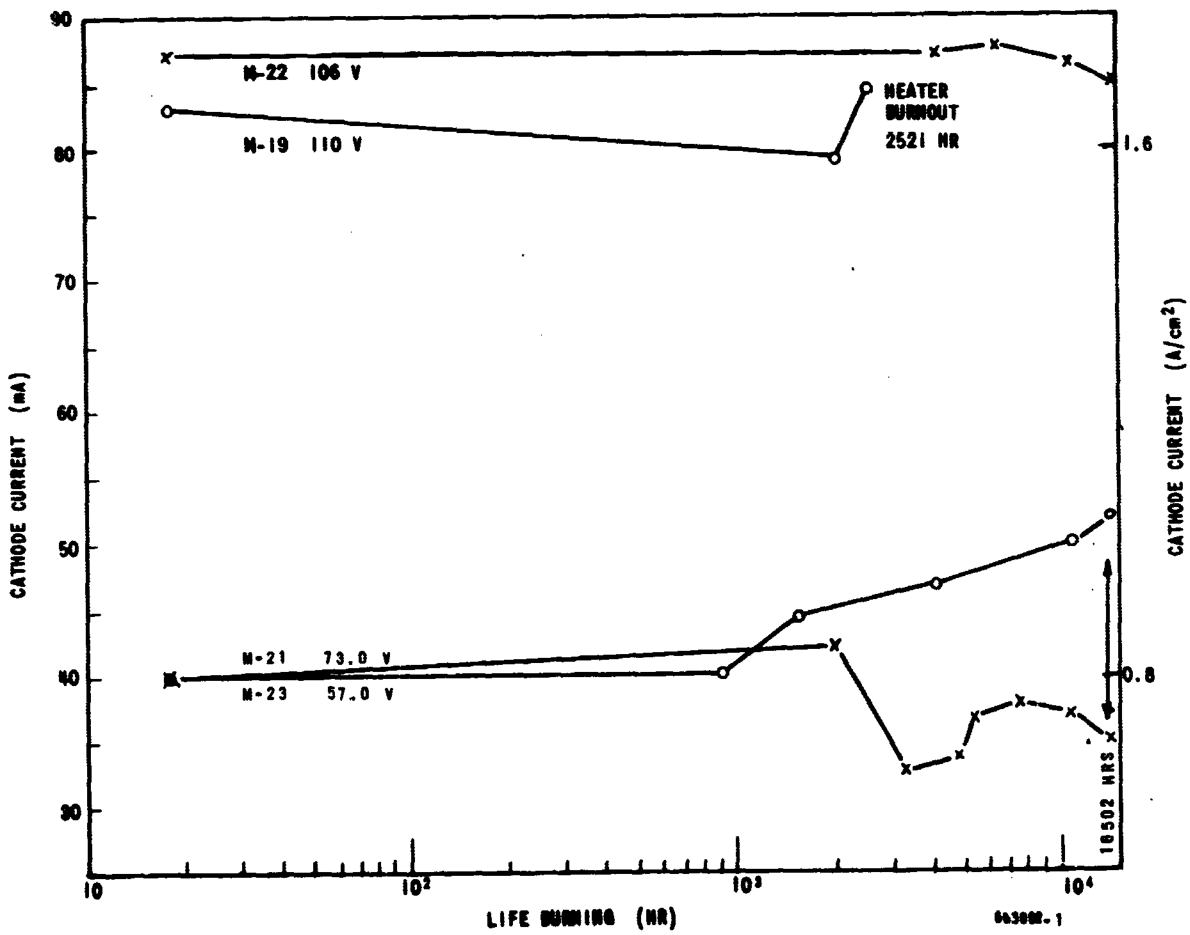

Figure 24. Pore-dispenser Cathode - T4 = 1100°C_{BR}

TABLE 9
LIFE TEST RESULTS
PORE-TYPE DISPENSER CATHODES

Test	Diode	Hours	I_p	I_p(-20%V)	I_p(+20%V)	Dip T° C	I_p@95%T
T1 - 950° C 0.2 A/cm^2	M-1 Ef=10.2V Ep=39V	0	10.0	8.4	12.0	880	8.80
		2688	11.0	8.9	13.2	891	8.00
		8693	11.0	9.0	12.9	904	8.57
		11046	11.0	9.0	13.1	891	8.69
		14423	11.8	9.9	14.0	910	8.63
		16805	11.4	9.4	13.8	925	7.81
				(32 V)	(46 V)		
	M-4 Ef=10.2V Ep=26V	0	10.0	8.3	12.5	888	8.81
		2688	10.0	8.4	12.2	906	8.25
		8693	9.9	8.4	12.0	898	7.11
		11046	9.9	8.2	11.9	904	7.87
		14423	10.7	9.2	13.2	855	9.22
		16805	10.9	9.0	13.0	910	7.50
				(22 V)	(32 V)		
T1 - 950° C 0.4 A/cm^2	M2 Ef=10.2V Ep= 9V	0	20.0	15.1	27.3	916	19.1
		2688	21.2	16.1	25.0	896	17.5
		8693	20.0	15.9	23.2	893	17.3
		11046	20.1	16.0	23.9	895	17.6
		14423	20.2	15.9	25.4	860	18.1
		16805	19.8	15.0	23.0	919	16.5
				(39 V)	(59 V)		
	M3 Ef=10.2V Ep=35V M-12	0	20.0	16.5	27.0	897	15.0
		2688	20.7	16.3	25.2	907	16.6
		8693	20.0	15.8	23.4	919	15.8
		11046	20.2	16.0	23.9	880	17.6
		14423	21.0	16.5	26.4	880	18.2
		16805	20.4	16.2	25.5	913	16.9
				(28 V)	(42 V)		

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TABLE 10
LIFE TEST RESULTS
PORE-TYPE DISPENSER CATHODES

Test	Diode	Hours	I_p	I_p(-20%V)	I_p(+20%V)	Dip T° C	I_p@95%T
T2 - 985° C	M7	0	20.0	16.8	27.5	899	19.6
0.4 A/cm^2	Ef=10.2V	2688	20.0	15.8	24.4	957	16.6
	Ep=34.5V	8693	23.9	18.8	29.9	957	16.3
		11046	23.9	18.8	29.9	945	17.5
		14423	23.2	18.4	29.0	906	18.8
		16805	22.9	18.0	28.2	968	15.7
				(28 V)	(42 V)		
	M9	0	20.0	14.6	28.5	910	18.8
	Ef=10.2V	2688	22.5	15.9	29.1	935	17.7
	Ep=40V	8693	21.5	15.8	27.2	941	17.5
		11046	21.8	15.6	27.8	941	17.1
		14423	23.0	16.2	30.2	902	18.8
		16805	21.3	15.5	28.0	968	16.3
				(30 V)	(50 V)		
T2 - 985° C	M11	0	40.0	32.0	49.5	964	28.0
0.8 A/cm^2	Ef=10.2V	2688	37.5	30.8	45.8	979	30.3
	Ep=65V	8693	34.3	28.4	41.2	970	31.6
		11046	35.5	29.7	41.2	946	35.0
		14423	34.6	29.0	40.4	960	34.0
		16805	31.0	26.8	34.3	985	29.7
				(54 V)	(76 V)		
	M12	0	40.0	31.0	50.0	913	38.0
	Ef=10.2V	2688	37.0	29.2	45.0	957	32.0
	Ep=54V	8693	32.1	25.9	37.3	951	31.6
		11046	33.8	27.2	39.9	910	34.5
		14423	36.2	28.9	44.8	873	36.5
		16805	35.3	28.7	42.2	951	31.2
				(44 V)	(64 V)		

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TABLE 11
LIFE TEST RESULTS
PORE-TYPE DISPENSER CATHODES

Test	Diode	Hours	I_p	I_p(-20%V)	I_p(+20%V)	Dip T°C	I_p@95%T
T3 - 1035°C	M13	0	30.0	22.5	38.5	965	29.2
0.6 A/cm^2	Ef=10.2V	2688	30.0	23.9	39.8	961	26.4
	Ep=45V	8693	32.2	24.6	41.0	1001	25.8
		11046	32.2	24.2	40.1	972	24.2
		14423	33.0	25.0	41.9	895	26.8
		16805	32.7	25.0	40.7	986	27.2
				(36 V)	(54 V)		
	M18	0	30.0	21.5	38.0	949	29.2
	Ef=10.2 V	2688	30.0	23.0	37.5	1003	25.6
	Ep=48.5V	8693	23.0	24.9	40.0	1001	25.0
		11046	31.7	24.7	40.0	1020	25.4
		14423	33.1	26.4	41.2	974	24.8
		16805	33.1	26.4	41.2	1026	22.0
				(39 V)	(59 V)		
T3 - 1035°C	M17	0	60.0	45.0	78.2	993	55.5
1.2 A/cm^2	Ef=10.2V	2688	61.2	47.8	77.4	1020	51.6
	Ep=90 V	8693	62.2	49.1	75.8	1035	51.6
		11046	63.2	51.2	78.8	1024	51.2
		14423	62.2	50.9	78.7	1035	51.2
		16805	60.0	48.6	71.8	1035	51.2
				(72 V)	(108 V)		
	M14	0	60.0	44.5	69.0	995	56.0
	Ef=10.2V	2688	54.9	41.2	70.2	977	55.2
	Ep=98V	8693	53.8	40.2	67.9	980	55.2
		11046	53.7	40.1	68.9	999	54.8
		14423	64.0	45.6	80.0	927	53.6
		16805	60.7	43.2	75.8	1017	52.0
				(78 V)	(118 V)		

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TABLE 12

LIFE TEST RESULTS

PORE-TYPE DISPENSER CATHODES

Test	Diode	Hours	I_p	I_p(-20%V)	I_p(+20%V)	Dip T°C	I_p @ 95%T
		0	40.0	23.0	52.0	957	37.6
	M21	2521	46.4	28.8	59.5	1055	34.6
	Ef=10.2V	8580	51.8	31.4	64.0	1042	32.5
T4 - 1100°C	Ep=57 V	10943	50.2	31.0	63.0	1048	29.0
		14120	54.0	33.4	67.2	925	37.5
0.8 A/cm^2		16502	53.0	33.0	66.0	1065	30.1
				(43 V)	(81 V)		
		0	40.0	24.0	51.0	997	38.0
	M23	2521	37.2	23.9	45.8	1097	31.0
	Ef=10.2V	8580	35.9	23.9	42.3	1100	25.0
	Ep=73V	10943	37.0	24.8	46.2	1100	29.0
		14120	25.9	18.5	30.2	1027	32.2
		16502	31.5	21.6	35.6	1100	28.5
				(49 V)	(87 V)		
		0	80.0	59.0	100.0	1039	73.0
	M22	2521	86.5	71.7	110.0	1051	66.0
T4 - 1100°C	Ef=10.2V	8580	86.9	74.2	110.0	1100	62.0
	Ep=106V	10943	86.3	74.1	110.0	1100	64.2
1.6 A/cm^2		14120	83.0	71.0	110.0	985	75.0
		16502	86.9	75.0	105	1052	69.5
				(84 V)	(128 V)		
		0	80.0	61.0	94.0	1049	77.0
	M19	2576	80.2	61.3	100.0	1039	75.0
	Ef=10.2V	1297	80.0	62.4	98.0	1053	65.0
	Ep=110V	2009	79.2	62.8	98.5	1066	65.0
		2521	84.5	67.0	104.0	1075	61.0
		2713	HEATER BURNOUT				
				(89 V)	(132 V)		

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The life test results for the diodes under T1 conditions as shown in Table No. 9 do not show any large deviations in all the test parameters up to this point of life burning with the exception of dip temperatures. An increase of 3 to 45°C should be noted.

The diodes operating under T2 conditions (Table 10) show an emission slump up to 22.5% at 0.8A/cm^2. The dip temperature has risen from 21 to 69°C. At this point of life burning the diodes under T2 conditions are considered to be showing satisfactory emission levels and cathode characteristics.

The diodes operating under T3 conditions (Table No. 11) do not show any large deviations in test parameter with the exception of dip temperature (increase of 19 to 38°C). The three diodes operating under T4 conditions are considered satisfactory up to 16,502 hours of life burning. Diode No. M-19 showed a heater burn-out at 2,713 hr.

In summary it can be said that the pore-type dispenser cathode in the test diode has operated successfully for cathode emission characteristics for at least 16,502 hours of life burning with cathode loading conditions varying from 0.2 to 1.6 A/cm^2 and the cathode temperature varying from 950 to 1100°C (brightness). It should also be noted that the cathodes have shown large amounts of barium metal evaporation which has deposited on the inner surface of the glass envelope below the anode height.

7.4 Standard Barium-strontium Oxide Cathode

The test diodes with barium-strontium oxide cathodes operating under T1 and T2 conditions completed 13,359 hours of life burning. The test diodes under T3 and T4 conditions completed 15,679 hours of life burning.

The life burning results at specified temperatures and anode voltages are shown in Figures 25 (T1), 26 (T2), 27 (T3) and 28 (T4). Other life test measurements are shown in Tables 13 (T1), 14 (T2), 15 (T3), and 16 (T4).

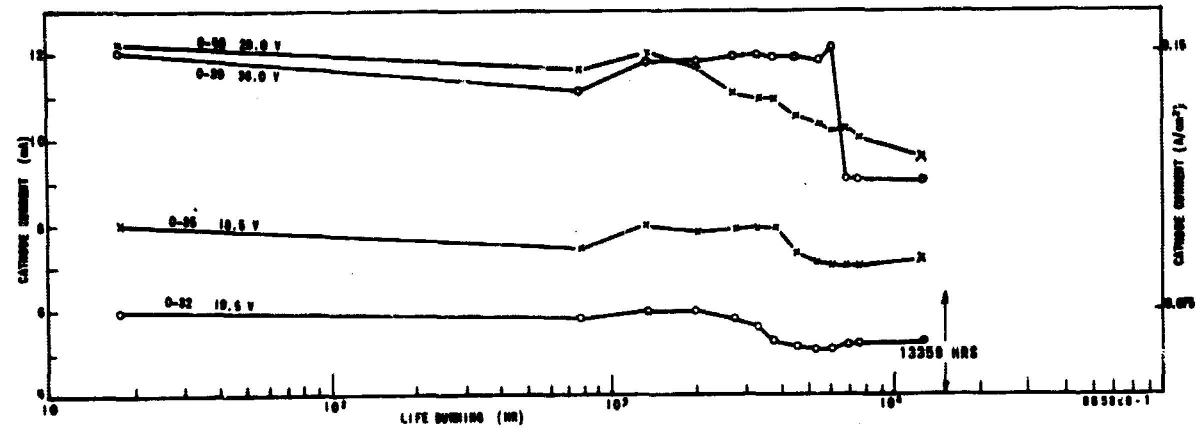

Figure 25. Standard Oxide Cathode - T1 = 800°C_{BR}

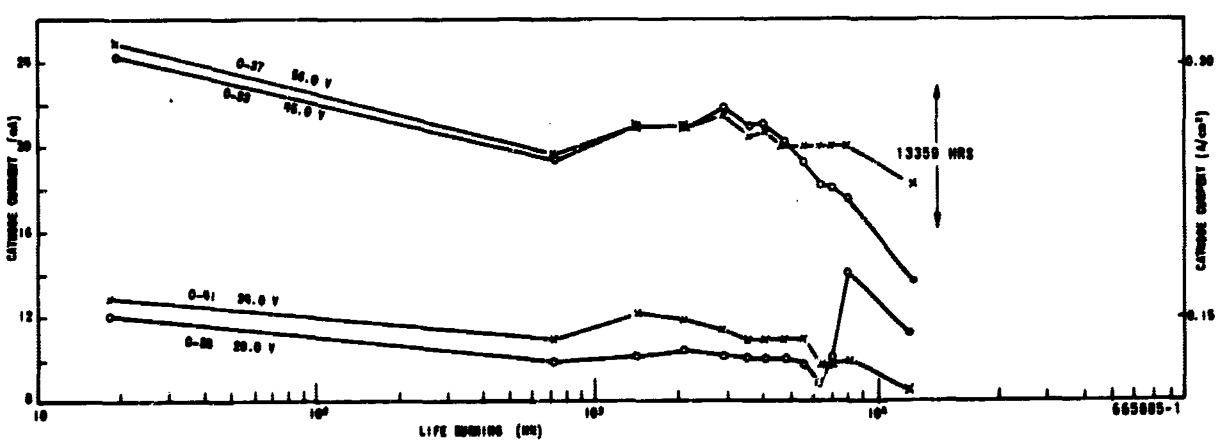

Figure 26. Standard Oxide Cathode - T2 = 825°C_{BR}

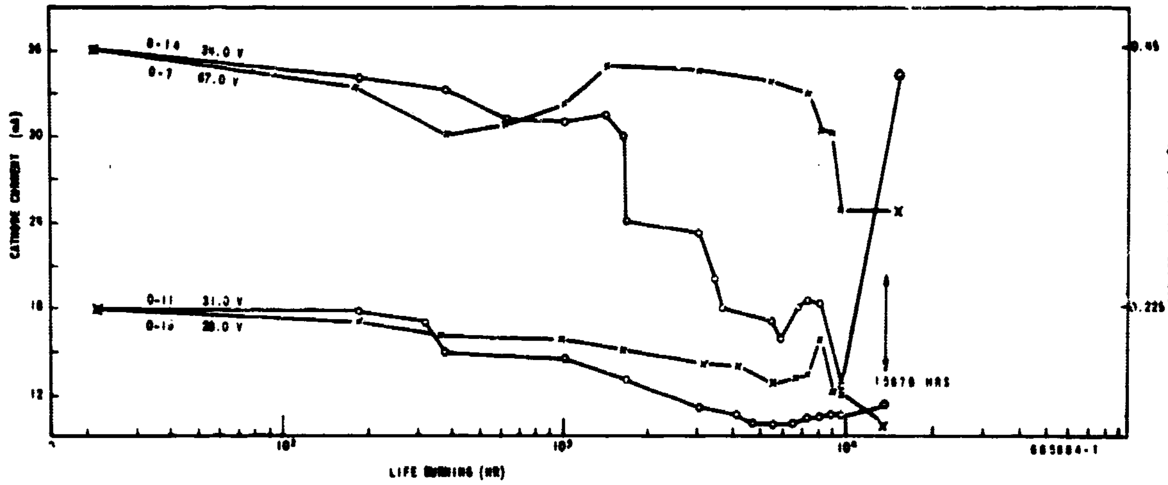

Figure 27. Standard Oxide Cathode - T3 = 825°C_{BR}

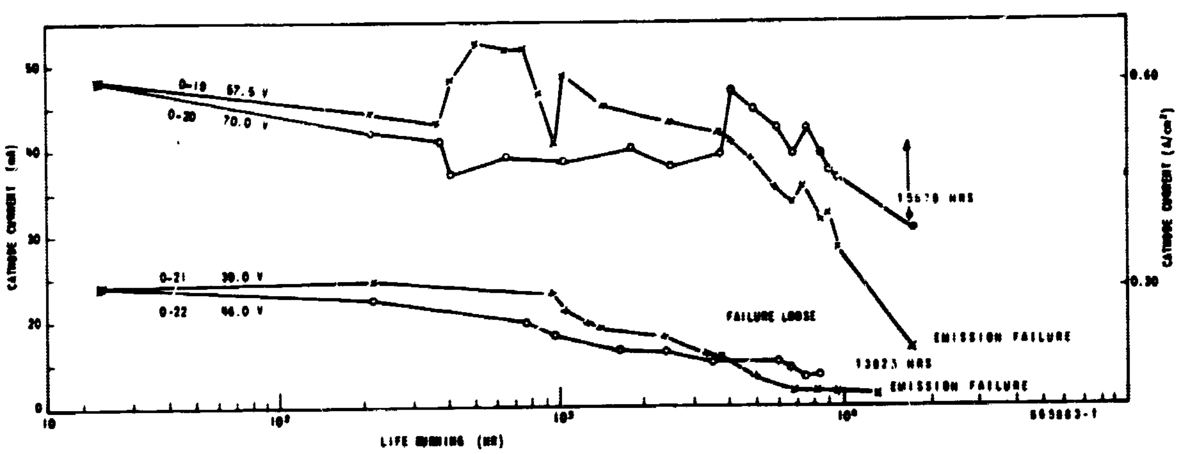

Figure 28. Standard Oxide Cathode - T4 = 850°C_{BR}

TABLE 13
LIFE TEST RESULTS
OXIDE-COATED CATHODES

Test	Diode	Hours	I_p	I_p(-20%V)	I_p(+20%V)	Dip T °C	I_p @ 95%T
T1 - 800°C 0.075 A/cm^2	O32 Ef=8.0V Ep=19.5V	0	6.0	4.7	7.9	722	4.13
		1371	6.0	4.9	7.4	666	5.14
		5238	5.1	4.2	6.1	693	5.19
		7600	5.2	4.2	6.0	732	4.95
		11605	5.5	4.7	6.1	760	4.88
		13359	5.5	4.7	6.2	788	4.88
				(15 V)	(24 V)		
	O35 Ef=8.0V Ep=18.5V	0	8.0	7.1	9.7	750	4.13
		1373	7.8	7.2	8.9	740	5.14
		5238	7.1	7.4	8.0	774	4.88
		7600	7.0	6.8	8.0	780	4.88
		11605	7.2	6.8	8.6	785	4.88
		13359	7.2	6.8	8.6	788	4.55
				(14 V)	(22 V)		
T2 - 825°C 0.15 A/cm^2	O39 Ef=8.0V Ep=36V	0	12.0	9.0	15.1	655	10.9
		1373	11.8	8.9	14.3	680	10.5
		5238	11.8	8.9	14.4	692	10.3
		7600	9.0	6.4	10.9	714	10.2
		11605	9.0	7.1	10.9	706	10.1
		13359	9.0	7.2	10.9	751	9.9
				(28 V)	(44 V)		
	O40 Ef=8.0V Ep=29V	0	12.0	9.6	14.7	769	9.3
		1373	12.0	9.9	14.1	703	10.1
		5238	10.3	8.9	12.2	743	9.1
		7600	10.0	8.4	12.0	757	9.8
		11605	9.0	7.8	10.9	766	9.8
		13359	9.0	7.6	10.8	785	9.3
				(23 V)	(35 V)		

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TABLE 14
LIFE TEST RESULTS
OXIDE-COATED CATHODES

Test	Diode	Hours	I_p(ma)	I_p(-20%V)	I_p(+20%V)	Dip T°C	I_p @ 95%T
	O38	0	12.0	9.3	15.2	741	11.0
	Ef=8.0V	1371	11.0	8.0	13.0	804	10.2
	Ep=29.0V	5238	9.9	8.0	11.4	825	8.7
T2 - 825°C		7600	14.2	7.4	10.6	825	9.7
		11605	11.4	10.1	12.9	825	7.7
0.15 A/cm^2		13359	11.1	10.0	12.6	825	6.0
				(23 V)	(35 V)		
	O41	0	12.0	9.1	14.7	727	11.0
	Ef=8.0V	1371	12.0	9.3	14.9	758	10.0
	Ep=34.0V	5238	11.0	8.5	13.2	825	10.1
		7600	10.0	7.9	12.1	825	9.7
		11605	8.5	7.0	10.4	825	9.5
		13359	8.4	7.0	10.0	825	9.0
				(27 V)	(41 V)		
	O33	0	24.0	19.0	30.4	787	21.0
	Ef=8.0V	1371	20.9	16.2	25.4	825	20.8
T2 - 825°C	Ep=45.0V	5238	19.2	15.0	23.0	825	18.0
		7600	16.9	14.2	20.2	825	17.2
0.30 A/cm^2		11605	13.5	11.2	15.8	825	16.8
		13359	12.9	10.8	14.9	825	13.9
				(36 V)	(54 V)		
	O37	0	24.0	19.1	30.7	737	22.6
	Ef=8.0V	1371	21.0	17.0	24.7	825	18.0
	Ep=56V	5238	20.0	16.5	23.5	825	19.1
		7600	20.0	16.9	23.3	825	20.1
		11605	21.0	17.2	28.3	781	21.3
		13359	18.0	15.0	21.9	825	16.0
				(45 V)	(67 V)		

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TABLE 15
LIFE TEST RESULTS
OXIDE-COATED CATHODES

Test	Diode	Hours	I_p(ma)	I_p(-20%V)	I_p(+20%V)	Dip T°C	I_p @ 95%T
T3 - 825°C 0.45 A/cm²	O7 Ef=8.0V Ep=34V	0	36.0	28.0	45.5	783	33.5
		3439	20.0	17.0	22.4	825	32.8
		7368	18.6	16.3	21.9	825	28.4
		9720	11.0	10.9	14.3	825	22.5
		13925	37.2	28.2	48.5	797	31.5
		15679	34.0	26.2	41.8	825	24.3
				(27 V)	(41 V)		
	O14 Ef=8.0V Ep=67V	0	36.0	28.0	44.5	768	31.7
		3439	35.4	27.0	46.2	825	29.3
		7368	33.0	26.9	44.2	825	24.8
		9720	29.8	24.2	42.5	825	28.4
		13925	27.2	22.6	41.5	825	23.6
		15679	25.0	21.4	39.2	825	27.0
				(54 V)	(80 V)		
T3 - 825°C 0.225 A/cm²	O11 Ef=8.0V Ep=31V	0	18.0	14.0	22.2	779	16.4
		3439	11.0	9.0	12.4	825	11.6
		7368	10.4	8.9	12.4	825	12.4
		9720	10.0	8.4	12.0	825	12.6
		13925	10.2	8.8	12.5	825	15.7
		15679	11.0	8.9	12.9	825	10.9
				(24 V)	(37 V)		
	O15 Ef=8.0V Ep=28V	0	18.0	13.9	23.5	769	16.6
		3439	14.2	11.3	18.0	825	13.5
		7368	13.4	10.7	15.9	825	10.8
		9720	12.4	11.8	19.8	825	11.9
		13925	12.2	10.0	14.2	825	15.7
		15679	12.8	10.0	14.0	825	10.1
				(22 V)	(34 V)		

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TABLE 16
LIFE TEST RESULTS
OXIDE-COATED CATHODES

Test	Diode	Hours	I_p(ma)	I_p(-20%V)	I_p(+20%V)	Dip T°C	I_p @ 95%T
T4 - 850°C 0.3 A/cm^2	O21 Ef=8.0V Ep=39V	0	24.0	18.2	29.0	774	21.6
		3439	15.0	12.2	19.8	850	18.3
		7368	13.5	10.9	17.4	850	16.9
		8138	13.9	10.9	16.3	850	16.4
		8933	DIODE FAILED - LOOSE ANODE				
				(29 V)	(46 V)		
	O22 Ef=8.0V Ep=46V	0	24.0	19.7	28.0	775	18.2
		3439	15.8	13.1	21.2	850	19.3
		7368	11.7	10.0	13.1	850	13.5
		9720	11.5	9.9	12.3	850	8.2
		13297	8.6	7.5	8.9	NO READING	
		13925	EMISSION FAILURE				
				(37 V)	(55 V)		
T4 - 850°C 0.6 A/cm^2	O19 Ef=8.0V Ep=57.5V	0	48.0	35.0	59.3	796	42.0
		3439	41.9	31.4	64.5	850	36.0
		7368	42.2	32.2	55.8	850	35.1
		9720	36.5	29.3	51.8	850	28.5
		13925	35.2	28.8	47.0	850	25.8
		15679	29.5	24.9	47.8	850	34.2
				(46 V)	(70 V)		
	O20 Ef=8.0V Ep=70V	0	48.0	36.8	60.0	769	42.6
		3439	41.4	32.0	55.3	850	37.5
		7368	35.8	26.9	44.9	850	31.2
		9720	28.3	23.1	42.7	850	24.2
		13925	25.9	22.2	29.0	850	22.5
		15679	12.0	11.9	14.0	850	25.2
				(53 V)	(81 V)		

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The diodes operating under T1 conditions at 800°C_{Br} cathode temperature have completed 13,359 hours of life burning with the diodes operating at 0.15 A/cm^2 showing a 25% slump in cathode emission. The dividers have shown a change of 38 to 96°C in dip temperature.

The diodes operating at T2 conditions have shown an emission slump of up to 43% with the diodes at the higher current density showing the greatest slump. It should be noted that the diodes at 0.15 A/cm^2 and 825°C cathode temperature had a dip temperature of 825°C at 5,238 hr as compared to the diodes under T1 conditions (0.15 A/cm^2 and 800°C cathode temperatures) which at 13,359 hours are still showing spare charge currents. The diodes at 0.30 A/cm^2 showed a dip temperature of 825°C at 1,371 hr of life burning.

The diodes at T3 conditions at 15,679 hours of life burning show approximately a 30% slump in cathode emission with the exception of diode No. 0-7 which is showing a very erratic behavior of decay and recovery of cathode emission. All the diodes show a dip temperature of 825°C at 3,439 hr.

The diodes operating at T4 conditions at 850°C_{Br} cathode temperature have shown three emission failures from 13,925 to 15,679 hours. Two of the failures were due to cathode emission and one was due to mechanical reasons. The first diode operating at 0.6A/cm^2 has shown a 39% emission slump in 15,679 hr.

It should be noted that the dip temperature reaches 850°C at 3,439 hr of life burning.

In summary, it should be noted that two diodes out of sixteen have failed because of cathode emission capabilities. These two diodes were operating under T4 conditions (highest temperature 850°C) and current density 0.3 - 0.6 A/cm^2). All the diodes under test showed signs of slumping emission when operated above 800°C or 0.15 A/cm^2.

Under the conditions of test, the diodes are capable of operating for at least 13,359 hr under the varying conditions noted in Table 1.

The dip temperature reaches the operating from 3,439 hours to 5,238 hr when operating above 800°C or 0.15 A/cm^2.

8.0 FURTHER TESTS WITH BARIUM-STRONTIUM OXIDE AND COATED PARTICLE CATHODES

Further tests were conducted with cathodes made from these different nickel cathode alloys and barium-strontium oxide and coated particle cathode coatings under Modification No. 1 of this study.

The life test conditions for various combinations of cathode alloys and coatings are shown in Table 17.

The total number of diodes involved in the tests was twenty. Three different cathode alloys (220, cathalloy A-33 and 0.1% zr in Ni-Pure nickel) were used with the Raytheon coating mixture C51-3 and two different cathode alloys (cathalloy-A33 and 0.1% zr in Ni-Pure nickel) were used with coated particle cathodes.

The cathodes were machined to the specifications noted in Section 3.2, Standard Barium-Strontium Oxide cathode (Area = 0.079 cm^2). After cleaning, the cathodes were vacuum fired at 1000°C for 30 minutes in a vacuum = 10^{-8} torr. The cathodes were then sprayed with either C51-3 or coated particle coating mixtures to a thickness of 0.0025 ± 0.005 inches with a density of 1.0 gm/cm^2. The cathodes were mounted into the diode test structure as noted in Section 4.0, The Diode Test Vehicle.

The diodes, using the prescribed cathodes (Table No. 17), were exhausted on a system consisting of a Vac-Ion pumping system (125 ℓ/s) for internal pumping of the vehicle and a vacuum bakeout oven backed by an oil diffusion pump capable of 1 x 10^{-6} torr pressure at 400 ℓ/s. The diodes were baked out at 450°C for 24 hr and then were processed according to Table No. 3, Exhaust processing of coated particle cathodes.

All the diodes at test proved to be very poor for cathode emission (max 12 mA at 50 V dc at 800-825°C_{Br}). A physical examination of the five lots of diodes showed the cathode coating to ve very thin and peeling from the nickel alloy base metal. The tests were discontinued at this time because of the cathode peeling problem.

TABLE 17
LIFE TEST PROCEDURES
MODIFICATION NO. 1

CATHODE	LIFE TEST TEMP.	REQ'D UNITS	CURRENT DENSITY ma/cm^2
Oxide Cathode	T_2	1	150
Using 220 Alloy	T_2	1	300
Nickel Base	T_3	1	225
(4 Units)	T_3	1	450
Oxide Cathode	T_2	1	150
Using Cathalloy	T_2	1	300
A-33 Nickel Base	T_3	1	225
(4 Units)	T_3	1	450
Oxide Cathode	T_2	1	150
Using 0.1% Zr in	T_2	1	300
Ni-pure Nickel Base	T_3	1	225
(4 Units)	T_3	1	450
Coated Particle	T_2	1	275
Cathode Using Cath-	T_2	1	550
alloy A-33 Nickel Base	T_3	1	415
(4 Units)	T_3	1	830
Coated Particle	T_2	1	275
Cathode Using 0.1%	T_2	1	550
Zr in Ni-pure Nickel Base	T_3	1	415
(4 Units)	T_3	1	830

New cathodes were prepared with the addition of a nickel powder underlay between the cathode coating and the base metal. The porous layer was made by spraying powdered mond nickel to a thickness of 0.001 - 0.003 inch and then sintering at 1140°C in wet hydrogen for 10 minutes. The cathodes were then mounted in diode structures and processed as described above.

The diodes (5 lots) were aged for 24-100 hr at 800-825 °C cathode temperature at an anode voltage of 50 V dc. The diodes at test showed the dip temperatures to be the same as the operating temperatures for given current loadings.

The test diodes were selected and placed on life burning with the highest possible cathode current that they would operate it in the space charge region (Table 17).

The life test results for coated particle cathodes are shown in Table 18. The cathodes show higher than usual slumps for 2000 hr of burning with no apparent dip temperature difference from the operating temperature. The life test results for the oxide coated cathodes are shown in Table 19. All the diodes show cathode failures at early periods of life burning (1,400 hours).

The main differences between these diodes and the diodes described in section 6.3 (the barium-strontium oxide cathode) are the cathode alloys and the methods of exhaust processing. The cathodes were vacuum fired at 1000°C and a nickel underlay was added to the cathode surface. The cathodes were exhaust processed by a slow heating schedule at coating decomposition by controlling the level of pump pressure at 10^{-7} torr. The previous diodes were exhausted by heating the cathodes rapidly, disregarding the pump pressure.

The coating particle cathodes had the same difference in manufacture but the exhaust schedule was the same as for the previous diodes.

The diodes behaved as did the coated particle cathode (section 6.4).

TABLE 18

LIFE BURNING - COATED-POWDER CATHODES

Test	Diode	Hours	I_p(ma)	Volts	I_p(± 20%V)
T2 - 850°C CPC with 0.1% Zr in NiPure	N-16	0	12.0	52	9.4-14.6
	0.15A	1463	10.9		9.0-12.8
	/cm^2	2110	10.0		9.0-12.9
	N-26	0	24.0	25	19.0-28.0
	0.30A	1463	16.8		14.5-18.1
	/cm^2	2110	14.8		13.0-16.4
T3 - 850°C CPC with 0.1% Zr in NiPure	No. 13	0	18.0	72	13.9-20.5
	0.225A	1463	10.3		8.7-12.9
	/cm^2	2110	11.8		9.9-14.7
	N-17	0	36.0	57	26.6-43.0
	0.45A	1463	23.3		20.0-26.8
	/cm^2	2110	22.0		19.5-25.1
T2 - 825°C CPC with A33 Nickel	N-21	0	19.0	38	17.0-21.8
	0.275A	1765	18.9		16.0-20.9
	/cm^2	2409	13.6		15.8-17.5
	N-31	0	37.0	61	29.8-48.9
	0.55A	1765	25.2		20.9-30.0
	/cm^2	2409	23.9		20.0-28.4
T3 - 825°C CPC with A33 Nickel	N-4	0	33.0	15	30.4-49.5
	0.415	1765	22.4		18.1-27.2
	/cm^2	2409	23.0		18.9-27.9
	N66	0	66.0	49	54.2-86.2
	0.830A	1765	24.8		21.5-28.6
	/cm^2	2409	22.2		19.2-26.0

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TABLE 19

LIFE BURNING - OXIDE-COATED CATHODES

Test	Diode	Hours	I_p(ma)	Volts	I_p(± 20%V)
T2 - 825°C Oxide with 0.1% Zr in NiPure	No. 3	0	24.0	29	19.0-29.2
	0.30A	1417	4.0		3.8-4.2
	/cm^2	2016	3.3		3.1-3.3
	No. 1	0	12.0	38	10.2-13.9
	0.15A	1417	3.3		3.1-3.3
	/cm^2	2016	3.2		3.2-3.2
T3 - 850°C Oxide with 0.1% Zr in NiPure	No. 2	0	18.0	38	15.4-26.2
	0.225A	1440	3.5		3.3-3.7
	/cm^2	2084	3.2		3.2-3.2
	No. 4	0	36.0	35	28.5-51.7
	0.45A	1440	3.5		3.4-3.5
	/cm^2	2084	3.5		3.2-3.5
T2 - 825°C Oxide with A33 Nickel	No. 11	0	24.0	58	18.0-30.6
	0.30A	1463	5.7		5.0-6.0
	/cm^2	2110	4.6		4.2-5.1
	No. 22	0	12.0	36	9.5-13.9
	0.15A	1463	7.2		7.2-8.4
	/cm^2	2110	6.8		6.2-7.4
T3 - 850°C Oxide with A33 Nickel	No. 24	0	18.0	36	14.0-22.4
	0.225A	1463	3.9		3.6-4.0
	/cm^2	2110	3.8		3.6-4.1
	No. 12	0	36.0	45	27.8-44.8
	0.45A	1463	6.2		5.9-7.0
	/cm^2	2110	5.2		4.9-5.6

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TABLE 20 (Cont'd)

LIFE BURNING - OXIDE-COATED CATHODES

Test	Diode	Hours	I_p(ma)	Volts	I_p(± 20%V)
T3 - 850°C Oxide with 220 Nickel	No. 5 0.225A /cm^2	0 1378 2022	18.0 2.8 2.9	34	14.2.24.8 2.7-2.9 2.7-2.9
	No. 1 0.45A /cm^2	0 1378 2022	36.0 2.7 2.9	65	28.4-41.5 2.6-2.7 2.7-2.9

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9.0 CONCLUSIONS AND RECOMMENDATIONS

In summarizing the results of this study of the life-burning capabilities of three different thermionic emitters, the following results are noted from this investigation of obtaining 1.0 A/cm^2 under dc voltage conditions at the lowest possible cathode temperature.

1. The pore-type dispenser cathode in this particular test diode has operated successfully for cathode emission characteristics for at least 16,502 hours of life burning with cathode current varying from 0.2 to 1.6 A/cm^2 and the cathode temperature varying from 950 to 1100°C (brightness). It should also be noted that the cathodes have shown large amounts of barium metal evaporation which is evident on the inner surface of the glass envelope of the diode below the anode height.

2. The standard oxide cathodes in the test diode have been operating from 13,925 to 15,678 hours under varying cathode current loading from 0.075 to 0.6 A/cm^2 and cathode temperature variations from 800°C to 850°C (brightness). During this period of life burning, the diodes have shown increases in slumping emission during life burning above 0.15 A/cm^2 and 800°C. The emission slump increases as the current loading is increased up to 0.6 A/cm^2 and also with increases in cathode temperature up to 850°C. A gradual decrease in life expectancy for the standard oxide cathodes can be noted in Figures 25-28 with two emission failures noted at 850°C$_{BR}$ (0.3 and 0.6 A/cm^2) at 10,000 hours.

3. The coated particle cathode, under the conditions of construction and test showed the capability of operating at 0.275 A/cm^2 at 850°C and 0.55 A/cm^2 at 900°C. A comparison of pulsed current capabilities of the oxide and coated particle cathode showed both cathodes capable of at least 1.0 A/cm^2 peak current at 350°C with the perveance slope being equal to 1.23 to 1.3 in both cases. The coated particle cathodes were not life tested because of the necessary high cathode temperature required for current loading conditions.

4. The exploratory tests described in Section 8.0 using three different cathode alloys with oxide and coated-particle cathodes showed very poor cathode emission capabilities and life expectancy. It should be noted that the main variable in these tests was the exhaust processing schedules. The cathode conversion cycle for the standard oxide cathodes during exhaust (Section 5.3) was very rapid as compared to the slow cathode conversion schedules used for these cathodes (Section 5.4).

9.1 Life Expectancy of Thermionic Emitters

Though the pore-type dispenser cathode (barium-aluminate type) has been used in microwave devices for approximately 15 years, very little published data is available for life burning characteristics of the cathode under varying current loading conditions and cathode temperatures.

In a study of the evaporation of barium from impregnated cathodes, Brodie and Jenkins[5] predict a life of 40,000 hours for a pore-type dispenser cathode which is 1 mm thick, 25% porous and operates at 1100°C. This prediction was made upon the basis of measured evaporation rates of barium from the cathode at 1100°C operation. Also a change in temperature of 120°C would change the evaporation rate by a factor of ten. If we consider the pore-type cathode used in these tests which has a porosity of 20% and a thickness equal to 0.045 in., the life expectancy of the cathode at 1100°C could be as high as 40,000 hours and at 950°C as high as 500,000 hours.

The standard oxide cathode which has been in use for at least 40 years has generally been operated under dc conditions at low current densities below 0.1 A/cm^2 at 800°C. Very little information is available for the life expectancy of oxide cathodes at conditions above 0.2 A/cm^2.

The writer conducted life burning tests with oxide cathodes[6] at 0.1 A/cm^2 with the cathode temperature at 800°C. The cathode used had a wall thickness of 0.003 in. and the vehicle was known as the A.S.T.M. standard diode. The diodes operated at these conditions for 60,000 hours and then showed a slump of 20-50% up to 78,134 hours at which time the test was discontinued. These tests covered a period of 10 years.

Kern, in his study of the life expectancy of oxide cathodes[7], predicts a life of 20 years for cathodes operating at 740°C with 0.2 A/cm^2. The cathodes have operated for 40,000 hours as of 1965.

The best estimate that can be made at this time for the life expectancy of the standard oxide cathode operating above 800°C and 0.2 A/cm^2 is from 20,000 to 50,000 hours with increased current density up to 0.6 A/cm^2.

In conclusion, it should be noted that the life expectancy of either the pore-type dispenser cathode or the standard oxide cathode is entirely dependent upon its gaseous environment within its vacuum enclosure.[8] Barium, which is the main factor contributing to thermionic emission in either cathode, is a very reactive metal, especially with gases such as hydrogen and carbon monoxide which have been identified as the predominant gases present in microwave devices.

The only possible way to achieve the theoretical life expectancy of the two forementioned cathodes is to operate the cathodes in vacuum under the cleanest and lowest possible gas pressure that can be attained.

9.2 Recommendations

In view of the findings of this study, the following recommendations are made for future consideration.

1. The pore-type dispenser cathode which shows the capability of the highest emission levels (1.6 A/cm^2) of the three cathodes evaluated in this study has the detrimental factor of high barium evaporation at operating temperatures ($1100^\circ C_{BR}$).

   A study should be made to reduce the high evaporation rate by either pre-evaporation or diffusion barrier techniques.

2. The study of oxide and coated particle cathodes should be continued in a diode or vehicle to eliminate the effects of anode poisoning which is predominant in this type of test vehicle. Some approaches to the problem of anode poisoning in the presence of oxide and coated particle cathodes are as follows:

   a. Use of water cooled anodes
   b. Selection of anode materials with prescribed processing to eliminate the effects of gas poisoning
   c. Geometric consiruction of vehicles to eliminate line-of-sight gas poisoning.

3. The chemistry and thermionic emitting properties of the oxide cathode should be evaluated with respect to the effect of exhaust processing (fast vs slow conversion).

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APPENDIX I

Procedure for Chemical Cleaning
of All Diode Parts.

APPENDIX I

Cleaning of Tantalum Parts

Table 1

Purpose

To specify a method for cleaning tantalum and tantalum parts prior to use.

Equipment

Acid-resistant sink with ventilating hood and hot and cold running water.
Polyethylene container of suitable size for acid solution.
Stainless steel baskets or tongs, preferably coated with a teflon type plastic.
Clean filtered compressed air.

Materials

Hydrofluoric Acid. (concentrate reagent grade)
Nitric Acid. (concentrate reagent grade)
Methanol.
Lint-free paper.

Procedure

1. Determine capacity of container by measuring (use a liter graduate) the amount of water necessary to fill it. Then pour 1/3 of this amount into empty container and mark container to indicate 1/3 volume of container. Add another 1/3 of amount necessary to fill container and mark 2/3 volume. Fill to 1/3 mark with water, add nitric acid to 2/3 volume mark, then fill with hydrofluoric acid.
2. Immerse parts for 5 seconds with gentle agitation.
3. Hold parts in fumes just above solution for 10 seconds.
4. Rinse in running cold tap water.
5. Repeat #2, 3 and 4 twice more.
6. Rinse thoroughly in running cold tap water.
7. Rinse in methanol.
8. Dry in stream of clean dry compressed air.
9. Wrap in lint-free paper.

Note

(1) Do not handle clean parts with fingers.
(2) Hydrofluoric acid and its containers should be handled wearing rubber gloves. Wash hands thoroughly with running water when through. Face shields shoud be worn while handling hydrofluoric solutions.
(3) Shop Manual "Handling corrosive materials" applies.

Cleaning of Nickel Parts

Table 2

Preparation of Solutions

1. Cleaning solution -

30% Hydrogen Peroxide	5 Parts
88% Formic Acid	10 Parts
Distilled water	80 Parts

Add the formic acid to the distilled water and then add the hydrogen peroxide.

2. Maintain bath temperature at 20°C - 25°C.

3. Change solution twice weekly.

Procedure

1. Place parts in suitable container or fixture.
2. Vaper degrease parts in permachlor.
3. Clean parts ultrasonically in Igepal solution.
4. Clean parts in solution for 8 - 9 minutes.
5. Rinse parts in overflowing tap water for 5 - 6 minutes.
6. Rinse and agitate parts in deionized water for 2 minutes minimum.
7. Rinse parts in deionized water for a minimum of 5 minutes.
8. Repeat step (7) twice, rinsing in the next cleanest tank each time.
9. Blow parts dry with filtered nitrogen.
10. Dry parts in nitrogen-fed oven (105°C-116°C) for 15 - 17 minutes.
11. Blow sample parts dry with filtered nitrogen. (Handle all parts with PVC palmed gloves).
12. Store parts in clean containers.

Table 3

Cleaning of Glass Parts

Preparation of Solution
Prepare 10% by volume - 30% hydrogen peroxide, 90% deionized water and add ammonium hydroxide to adjust pH to 11 (check with pH paper).
Procedure
a. Boil glass piece in solution for 30 minutes.
b. Rinse in overflowing tap water for 5 minutes.
c. Rinse in deionized water.
d. Dry in nitrogen-fed oven at 110°C for 15 minutes.